



QUALITY ASSURANCE PROJECT PLAN FOR REMOVAL PLAN (Revised)

**20 JEFFERSON AVENUE
ELGIN, ILLINOIS**

**Prepared for
Jefferson Yard Removal Action Group**

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REVISION 1**

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B	Field SOPs

QAPP Worksheet #1
Title and Approval Page

Site Name/Project Name: 20 Jefferson Avenue Removal Plan
Site Location: Elgin, Illinois

QUALITY ASSURANCE PROJECT PLAN FOR 20 JEFFERSON AVENUE REMOVAL PLAN (REVISED)
Document Title

USEPA - Region V.
Lead Organization

Grant Anderson/ Conestoga Rovers & Associates, Inc.
Preparer's Name and Organizational Affiliation

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Preparer's Address, Telephone Number, and E-mail Address

February 19, 2009
Preparation Date

Investigative Organization's Project Manager: _____
Signature
Steve Voss
Conestoga-Rovers & Associates, Inc.

Date

Investigative Organization's QA Manager: _____
Signature
Grant Anderson
Conestoga-Rovers & Associates, Inc.

Date

Lead Organization's Project Manager _____
Signature
Alan Bielawski
The Group

Date

QAPP Worksheet #1
Title and Approval Page (continued)

Site Name/Project Name: 20 Jefferson Avenue Removal Plan
Site Location: Elgin, Illinois

Project Manager

Signature

Steve Voss/CRA

Date

Approval Signatures:

Signature

Steve Faryan/USEPA - Region V

Date

Signature

Date

Other Approval Signatures:

Signature

Amy McCormick / Test America

Date

Document Control Number: _____

QAPP Worksheet #2
QAPP Identifying Information

Site Name/Project Name: 20 Jefferson Avenue
Site Location: Elgin, Illinois

Contractor Name: Conestoga-Rovers & Associates, Inc.

1. Identify guidance used to prepare QAPP:

Uniform Federal Policy for Quality Assurance Project Plans (Final – Version 1, March 2005)

2. Identify regulatory program: Office of Emergency Response

3. Identify approval entity: USEPA

4. Indicate whether the QAPP is a generic or a **project-specific QAPP**. (bold one)

5. List dates of scoping sessions that were held: Not applicable

6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Dated
<u>QAPP (revised) (CRA)</u>	<u>1994</u>

7. List organizational partners (stakeholders) and connection with lead organization:

USEPA: Regulatory Oversight

8. List data users: The Group, CRA, USEPA

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table.

Provide an explanation for their exclusion below: _____

QAPP Worksheet #2
QAPP Identifying Information (continued)

Note: QAPP elements and required information that are not applicable to the project are circled and an explanation is provided in the QAPP.

<i>Required QAPP Element(s) and Corresponding QAPP Section(s)</i>	<i>Required Information</i>	<i>QAPP Worksheet #</i>
PROJECT MANAGEMENT AND OBJECTIVES		
2.1 Title and Approval Page	- Title and Approval Page	1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	3 4
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	5 6 7 8

<i>Required QAPP Element(s) and Corresponding QAPP Section(s)</i>	<i>Required Information</i>	<i>QAPP Worksheet #</i>
2.5 Project Planning/Problem Definition 2.5.1Project Planning (Scoping) 2.5.2Problem Definition, Site History, and Background	<ul style="list-style-type: none"> - Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present) 	<div>9</div> <div>10</div>
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2Measurement Performance Criteria	<ul style="list-style-type: none"> - Site-Specific PQOs - Measurement Performance Criteria Table 	<div>11</div> <div>12</div>
2.7 Secondary Data Evaluation	<ul style="list-style-type: none"> - Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table 	<div>13</div>
2.8 Project Overview and Schedule 2.8.1Project Overview 2.8.2Project Schedule	<ul style="list-style-type: none"> - Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table 	<div>14</div> <div>15</div> <div>16</div>

QAPP Worksheet #2
QAPP Identifying Information (continued)

<i>Required QAPP Element(s) and Corresponding QAPP Section(s)</i>	<i>Required Information</i>	<i>QAPP Worksheet #</i>
Measurement/Data Acquisition		
3.1 Sampling Tasks	- Sampling Design and Rationale	17
3.1.1 Sampling Process Design and Rationale	- Sample Location Map	
3.1.2 Sampling Procedures and Requirements	- Sampling Locations and Methods/ SOP	18
3.1.2.1 Sampling Collection Procedures	- Requirements Table	
3.1.2.2 Sample Containers, Volume, and Preservation	- Analytical Methods/SOP	19
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3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures	- Field Quality Control Sample Summary Table	
3.1.2.5 Supply Inspection and Acceptance Procedures	- Sampling SOPs	21
3.1.2.6 Field Documentation Procedures	- Project Sampling SOP	
	- References Table	22
	- Field Equipment Calibration, Maintenance, Testing, and Inspection Table	
3.2 Analytical Tasks	- Analytical SOPs	
3.2.1 Analytical SOPs	- Analytical SOP	23
3.2.2 Analytical Instrument Calibration Procedures	- References Table	
3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures	- Analytical Instrument Calibration Table	24
3.2.4 Analytical Supply Inspection and Acceptance Procedures	- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	25

<i>Required QAPP Element(s) and Corresponding QAPP Section(s)</i>	<i>Required Information</i>	<i>QAPP Worksheet #</i>
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation - Handling, Tracking, and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal	26 27
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree	28
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs	29 30

QAPP Worksheet #2
QAPP Identifying Information (continued)

<i>Required QAPP Element(s) and Corresponding QAPP Section(s)</i>	<i>Required Information</i>	<i>QAPP Worksheet #</i>
Assessment/Oversight		
4.1 Assessments and Response Actions	- Assessments and Response Actions	
4.1.1 Planned Assessments	- Planned Project Assessments Table	31
4.1.2 Assessment Findings and Corrective Action Responses	- Audit Checklists - Assessment Findings and Corrective Action Responses Table	32
4.2 QA Management Reports	- QA Management Reports Table	33
4.3 Final Project Report		
Data Review		
5.1 Overview		
5.2 Data Review Steps	- Verification (Step I) Process Table	34
5.2.1 Step I: Verification		
5.2.2 Step II: Validation	- Validation (Steps IIa and IIb) Process Table	35
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5.2.3 Step III: Usability Assessment		
5.2.3.1 Data Limitations and Actions from Usability Assessment	- Usability Assessment	37
5.2.3.2 Activities		
5.3 Streamlining Data Review		
5.3.1 Data Review Steps To Be Streamlined		
5.3.2 Criteria for Streamlining Data Review		
5.3.3 Amounts and Types of Data Appropriate for Streamlining		

QAPP Worksheet #3
Distribution List

<i>QAPP Recipients</i>	<i>Title</i>	<i>Organization</i>	<i>Telephone Number</i>	<i>Fax Number</i>	<i>E-mail Address</i>	<i>Document Control Number</i>
Steve Faryan	Remedial Project Manager	U.S. EPA – Region V	312-353-9351		faryan.steven@epa.gov	
Alan Bielawski	Project Manager	The Group	312-853-7000	312-853-7036	abielawski@sidley.com	
Steve Voss	Project Manager	Conestoga-Rovers & Associates	651-639-0913	651-639-0923	svoss@croworld.com	
Grant Anderson	QA Manager/Data Validator	Conestoga-Rovers & Associates	651-639-0913	651-639-0923	ganderson@croworld.com	
Dorothy Leeson	Quality Manager	Test America North Canton, OH	330-497-9396	330-497-0772	dorothy.leeson@testamericainc.com	
Amy McCormick	Project Manager	Test America North Canton, OH	330-497-9396	330-497-0772	amy.mccormick@testamericainc.com	

QAPP Worksheet #4
Project Personnel Sign-Off Sheet

Note: Project Personnel sign-off sheets will be obtained by the CRA QA manager for key project personnel who have not already signed the approval page (Worksheet #1). The CRA QA Manager will ensure that these sheets are kept on file and they are obtained prior to each person beginning any of their project work.

Organization: Test America, Inc.

<i>Project Personnel</i>	<i>Title</i>	<i>Telephone Number</i>	<i>Signature</i>	<i>Date QAPP Read</i>
Dorothy Leeson	Quality Manager	330-497-9396		

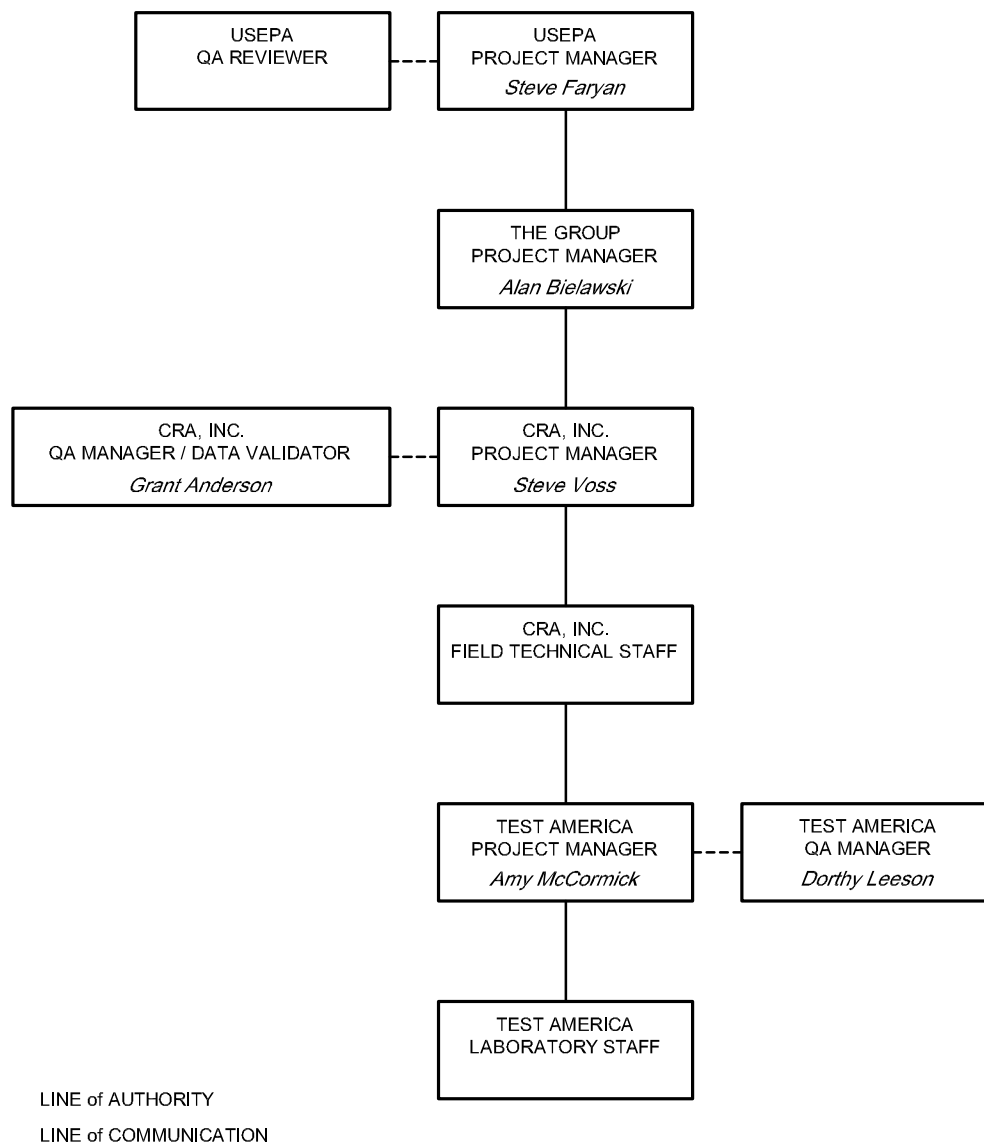


figure 5A

QAPP WORKSHEET #5
PROJECT ORGANIZATION CHART
20 JEFFERSON AVENUE - REMOVAL ACTION
Elgin, Illinois



QAPP Worksheet #6
Communication Pathways

<i>Communication Driver</i>	<i>Steps in Process</i>	<i>Timing</i>	<i>Means of Communication</i>
Amendment to the QAPP	1) The Group Project Manager (The Group PM) notifies all document holders of a pending amendment.	ASAP, but prior to conducting affected work	Telephone or e-mail
	2) The Group PM provides draft amendment to all document holders for review.	ASAP, but prior to conducting affected work	Hard copy by mail
	3) The Group PM coordinates comment resolution.	ASAP, but prior to conducting affected work	Telephone, e-mail, or meeting
	4) The Group PM coordinates amendment approval (signatures) of those persons who approved original QAPP and distributes amendment to document holders.	ASAP, but prior to conducting affected work	Hard copy by mail
Laboratory Deviation	1) Analyst notifies Laboratory Project Manager and QA Manager.	ASAP and within one business day	In person, by telephone, or e-mail
	2) QA Manager initiates Corrective Action Form.	Same day	Hard Copy
	3) Laboratory Project Manager (PM) notifies CRA QA Manager.	ASAP and within one business day	Telephone or e-mail
	4) CRA QA Manager notifies CRA PM who notifies The Group PM.	ASAP and within two business days	Telephone or e-mail
	5) The Group PM notifies EPA PM.	ASAP and within two business days	Telephone or e-mail
	6) Approval of the means of resolution by EPA PM.	ASAP	Telephone or e-mail

Field Work Deviation	1) Sampler notifies CRA PM and QA Manager and documents in field notes.	ASAP and within one business day	In person, by telephone, or e-mail
	2) QA Manager orders sampler to stop work (if deemed necessary).	Same day	In person, by telephone, or e-mail
	3) CRA PM notifies The Group PM.	ASAP and within two business days	Telephone or e-mail
	4) The Group PM notifies EPA PM.	ASAP and within two business days	Telephone or e-mail
	5) Approval of the means of resolution by EPA PM.	ASAP	Telephone or e-mail

Project Delay	1) CRA PM notifies The Group PM.	ASAP	Telephone or e-mail
	2) The Group PM notifies EPA PM.	ASAP	Telephone or e-mail
	3) Approval of the means of resolution by EPA PM.	ASAP	Telephone or e-mail

Laboratory Data Rejection	1) CRA QA Manager notifies CRA PM who notifies The Group PM.	ASAP and within two business days	Telephone or e-mail
	2) The Group PM notifies EPA PM.	ASAP and within two business days	Telephone or e-mail
	3) Approval of the means of resolution by EPA PM.	ASAP	Telephone or e-mail

Outlier Identification	1) CRA PM notifies The Group PM.	ASAP	Telephone or e-mail
	2) The Group PM notifies EPA PM.	ASAP	Telephone or e-mail
	3) Approval of the outlier identification by EPA PM.	ASAP	Telephone or e-mail
	4) CRA PM documents outlier identification in the Monitoring Report.	With report submittal	Hard Copy

Notes:

- 1) Notifications by The Group PM may also be delegated by The Group PM to the CRA PM, or CRA QA Manager.
- 2) Any QAPP modification or deviation in QAPP procedures (field or laboratory) must be documented in writing and approved by all signatories of the QAPP prior to conducting affected field work.

QAPP Worksheet #7
Personnel Responsibilities and Qualifications Table

<i>Name</i>	<i>Title</i>	<i>Organizational Affiliation</i>	<i>Responsibilities</i>	<i>Education and Experience Qualifications</i>
Steve Faryan	Remedial Project Manager	U.S. EPA – Region V	<ul style="list-style-type: none"> • Provide oversight of the entire site for work performed under this QAPP. • Review the QAPP and provide consistency, including subsequent revisions. • Coordination with USEPA QA Subcontractor • Coordinate collection of USEPA QA split samples, as deemed necessary by the USEPA. • Audit any field or laboratory work, as deemed necessary by the USEPA. • Review the data presentation and analysis in the Construction Completion Report. 	Note 1
Alan Bielawski	Project Manager	The Group	<ul style="list-style-type: none"> • Review and approve the QAPP, including subsequent revisions. • Coordinate with the CRA Project Manager and USEPA. 	Note 1
Steve Voss	Project Manager	CRA, Inc.	<ul style="list-style-type: none"> • Review and approve the QAPP, including subsequent revisions. • Coordinate with the QA Manager on all QA/QC matters. • Ensure compliance with the QAPP for all project work. • Assign trained staff and resources to complete work in accordance with the QAPP. • Coordinate preparation of the Construction Completion Report. 	Note 1

QAPP Worksheet #7
Personnel Responsibilities and Qualifications Table (continued)

<i>Name</i>	<i>Title</i>	<i>Organizational Affiliation</i>	<i>Responsibilities</i>	<i>Education and Experience Qualifications</i>
Grant Anderson	QA Manager/ Data Validator	CRA, Inc.	<ul style="list-style-type: none"> • Review and approve the QAPP, including subsequent revisions. • Conduct an annual field audit, and audit other field or laboratory work, as deemed necessary. • Coordinate with the external data validator • Prepare laboratory data validation reports. • Provide QA and data usability support to CRA, Inc. 	Note 1
Samplers	Field Technical Staff	CRA, Inc.	<ul style="list-style-type: none"> • Ensure QAPP-specified sample collection/handling/shipping procedures are followed. • Document sampling events and field observations. • Identify and report field problems to the Project Manager. 	Note 1
Dorothy Leeson	Quality Manager	Test America North Canton, OH	<ul style="list-style-type: none"> • Monitor and evaluate laboratory QA/QC activities, reporting deficiencies and identifying resource requirements to the Laboratory General Manager. • Conduct and document annual internal audits of laboratory procedures. • Review laboratory SOPs and laboratory Quality Assurance Manual/QAM (Appendix A). • Arrange the analysis of laboratory QC and performance evaluation samples. • Schedule and document annual Method Detection Limit (MDL) studies. • Maintain staff training records. • Maintain the laboratory corrective action program. • Review and approve the laboratory elements of this project QAPP. 	Note 1

<i>Name</i>	<i>Title</i>	<i>Organizational Affiliation</i>	<i>Responsibilities</i>	<i>Education and Experience Qualifications</i>
Amy McCormick	Project Manager	Test America North Canton, OH	<ul style="list-style-type: none"> • Ensure timely and accurate communication between CRA and laboratory. • Ensure that the requirements of the laboratory QAPP and the laboratory elements of this QAPP are implemented. • Ensure timely response to CRA requests for information and audits. • Perform final review of analytical data reports to ensure that all requirements are met. 	Note 1
	Quality Manager	U.S. EPA – Region V	<ul style="list-style-type: none"> • Identify and report problems to the Remedial Project Manager. • Review and approve the elements of this project QAPP. 	Note 1
Lab Staff		Test America North Canton, OH	<ul style="list-style-type: none"> • Ensure that the requirements of the laboratory QAPP and the laboratory elements of this QAPP are implemented. • Ensure timely response to CRA requests for information and audits. 	Note 1

Notes:

1) Resumes for each individual working on this project will be kept on file at each organization, and will be available to other organizations upon request.

QAPP Worksheet #8
Special Personnel Training Requirements Table

Note: Field staff must be trained in collecting valid samples while laboratory staff are trained in proper analytical procedures. Both field and lab staff must perform standard operating procedures, where applicable. The laboratory must be a NELAC/NELAP certified laboratory.

<i>Item</i>	<i>Training Procedures</i>	<i>Person Responsible for Training Documentation</i>	<i>Training Documentation Procedures</i>
Employee Orientation and Training	Refer to Test America SOP# CORP-QA-0013NC, Rev 1.2	Test America QA Manager	Refer to Test America SOP# CORP-QA-0013NC, Rev 1.2

QAPP Worksheet #9
PROJECT SCOPING SESSION PARTICIPANTS SHEET

Project Name <u>20 Jefferson Avenue Removal Action QAPP</u> Projected Date(s) of Sampling: <u>Spring 2009</u> Project Manager <u>Steve Voss</u>		Site Name <u>20 Jefferson Avenue</u> Site Location <u>Elgin, Illinois</u>		
Date of Session: October 7, 2008 Scoping Session Purpose: To discuss scope of removal action and USEPA expectations.				
Name	Affiliation	Phone #	E-mail Address	Project Role
Steve Faryan	U.S. EPA - Region V	312-353-9351	faryan.steven@epa.gov	Remedial Project Manager
Ron Frehner	CRA, Inc.	651-639-0913	rfrehner@croworld.com	Project Manager

Action Items: Prepare draft QAPP and Removal Plan and submit to USEPA by late February 2009.

Consensus Decisions:

- The QAPP will follow the Uniform Federal Policy (UFP) for QAPPs.
- The worksheet format described in UFP guidance will be used, making QAPP appearance quite different than prior QAPPs prepared for the site.
- 100% of data will be validated.
- QC samples will be similar to prior QAPPs prepared for the site.

QAPP Worksheet #10
(UFP-QAPP Manual Section 2.5.2)
Problem Definition

The problem to be addressed by the project:

PCB, dioxin, lead, cadmium, TCLP lead, and TCLP cadmium impacted soil present at the Site exceeding USEPA soil remedial objectives.

The environmental questions being asked:

Following soil removal activities, if additional soil sampling indicates that soil exceeds remedial objectives, then additional soil removal will be conducted until soil remedial objectives are met.

Observations from any site reconnaissance reports:

The Site displays no obvious signs of soil contamination.

A synopsis of secondary data or information from site reports:

Pre-removal investigation data indicates several areas where the site soil exhibits parameter concentrations exceeding remedial objectives. The investigation results provide a spatial delineation of most of the areas of impacted soil, allowing for soil removal without the need for follow-up sampling.

The possible classes of contaminants and the affected matrices:

PCB, lead, cadmium, dioxin in soil.

The rationale for inclusion of chemical and nonchemical analyses:

Soil sampling will be used, as necessary, to determine levels of site contaminants present in the remaining soils following soil removal.

Information concerning various environmental indicators:

Decisions regarding whether further excavation will be needed are based upon the previous investigation data results or, if collected, additional soil samples collected following excavation.

Project decision conditions ("If..., then..." statements):

If the confirmatory sampling data demonstrates that the remedial objectives have been met, no additional samples will be needed and the area will then be deemed "clean".

If the confirmatory sampling data indicate that the remedial objectives have not been met, then further excavation will be conducted until the remedial objectives are achieved.

QAPP Worksheet #11
(UFP-QAPP Manual Section 2.6.1)
Project Quality Objectives/Systematic Planning Process Statements

Who will use the data?

The data will be used by CRA, the Group, and USEPA.

What will the data be used for?

The data will be used to determine whether the project remedial objectives have been achieved.

What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)

Confirmatory soil samples will be collected from the soil excavation areas for the analysis of PCBs, total lead, total cadmium, TCLP lead, and TCLP cadmium. All collected samples will be analyzed by an off-site laboratory. No field screening will be conducted.

How "good" do the data need to be in order to support the environmental decision?

If collected, all soil data will be validated.

How much data are needed? (number of samples for each analytical group, matrix, and concentration)

Approximately 20 confirmatory soil samples will be collected.

Where, when, and how should the data be collected/generated?

Following soil excavation, sidewall samples (mid-height) will be collected at intervals of no greater than 25 feet and excavation bottom samples would be collected on an approximate 25-foot grid pattern.

Who will collect and generate the data?

CRA will collect the confirmatory soil samples to be analyzed by a subcontracted laboratory.

How will the data be reported?

All collected data will be reported to USEPA during the project, prior to backfilling of the excavation areas. The final analytical data will be documented in the Construction Completion Report.

How will the data be archived?

Collected soil data will be archived electronically by CRA

QAPP Worksheet #12

Measurement Performance Criteria Table

<i>Matrix</i>	<i>Soil</i>				
<i>Analytical Group</i>	<i>PCBs</i>				
<i>Concentration Level</i>	<i>Medium</i>				
<i>Sampling Procedure¹</i>	<i>Analytical Method/SOP²</i>	<i>Data Quality Indicators (DQIs)</i>	<i>Measurement Performance Criteria³</i>	<i>QC Sample and/or Activity Used to Assess Measurement Performance</i>	<i>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)</i>
SOP-1	SW846: 8082 (NC-GC-038-rev.0)	Precision	RPD \leq 50% (or $\pm 2 \times$ RL if sample or duplicate is $< 5 \times$ RL)	Field Duplicates	S & A
		Accuracy & Precision	% Recoveries and RPD as follows: Aroclor 1016 10-199 (30)	MS/MSD ⁴	S & A
		Accuracy	No Detections exceeding the RL	Method Blank, Rinsate Blank	A (Method blk), S & A (Rinsate blk)
		Accuracy	% Recoveries as follows: Decachlorobiphenyl 10-199 TCMX 10-196	Surrogates	A
		Accuracy	% Recoveries and RPD as follows: Aroclor 1016 34-127 (30)	Laboratory Control Sample	A
		Accuracy	RPD \leq 50% (or $\pm 2 \times$ RL if sample or duplicate is $< 5 \times$ RL)	QA split sample ⁵	S&A
		Sensitivity	RLs \leq Half the Action Level	RL Adequacy Check	A
		Field Completeness	100%	Data Completeness Check	S
		Analytical Completeness	100%	Data Completeness Check	A

<i>Matrix</i>	<i>Soil</i>				
<i>Analytical Group</i>	<i>Dioxins</i>				
<i>Concentration Level</i>	<i>Medium</i>				
<i>Sampling Procedure¹</i>	<i>Analytical Method/SOP²</i>	<i>Data Quality Indicators (DQIs)</i>	<i>Measurement Performance Criteria³</i>	<i>QC Sample and/or Activity Used to Assess Measurement Performance</i>	<i>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)</i>
SOP-1	SW846: 8280A (WS-ID-011-rev 4)	Precision	RPD \leq 50% (or $\pm 2 \times$ RL if sample or duplicate is $< 5 \times$ RL)	Field Duplicates	S & A
		Accuracy & Precision	% Recoveries and RPD as follows: 2378-TCDD 71-131 (25) 12378-PeCDD 65-140 (25) 123478-HxCDD 67-145 (25) 123678-HxCDD 57-144 (25) 123789-HxCDD64-134 (25) 1234678-HpCDD 66-133 (25) OCDD 61-135 (25)	MS/MSD ⁴	S & A
		Accuracy	No Detections exceeding the RL	Method Blank, Rinsate Blank	A (Method blk), S & A (Rinsate blk)
		Accuracy	13C-2378-TCDD 25-150 13C-123678-HxCDD 25-150 13C-OCDD 25-150	Internal Stds	A
		Accuracy	% Recoveries and RPD as follows: 2378-TCDD 71-131 (25) 12378-PeCDD 65-140 (25) 123478-HxCDD 67-145 (25) 123678-HxCDD 57-144 (25) 123789-HxCDD64-134 (25) 1234678-HpCDD 66-133 (25) OCDD 61-135 (25)	Laboratory Control Sample	A

<i>Matrix</i>	<i>Soil</i>				
<i>Analytical Group</i>	<i>Dioxins</i>				
<i>Concentration Level</i>	<i>Medium</i>				
<i>Sampling Procedure¹</i>	<i>Analytical Method/SOP²</i>	<i>Data Quality Indicators (DQIs)</i>	<i>Measurement Performance Criteria³</i>	<i>QC Sample and/or Activity Used to Assess Measurement Performance</i>	<i>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)</i>
		Accuracy	RPD \leq 50 % (or $\pm 2 \times$ RL if sample or duplicate is $< 5 \times$ RL)	QA split sample ⁵	S&A
		Sensitivity	RLs \leq Half the Action Level	RL Adequacy Check	A
		Field Completeness	100%	Data Completeness Check	S
		Analytical Completeness	100%	Data Completeness Check	A

<i>Matrix</i>	<i>Soil</i>				
<i>Analytical Group</i>	<i>Total, TCLP Metals</i>				
<i>Concentration Level</i>	<i>Medium</i>				
<i>Sampling Procedure¹</i>	<i>Analytical Method/SOP²</i>	<i>Data Quality Indicators (DQIs)</i>	<i>Measurement Performance Criteria</i>	<i>QC Sample and/or Activity Used to Assess Measurement Performance</i>	<i>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)</i>
SOP-1	SW846: 1311/6010 (NC-MT-012-rev.1, NC-OP-033)	Precision	RPD \leq 50% (or $\pm 2 \times$ RL if sample or duplicate is $< 5 \times$ RL)	Field Duplicates	S & A
		Accuracy & Precision	% Recoveries and RPD as follows: Cadmium 75-125 (20) Lead 75-125 (20) TCLP Cadmium 50-150 (20) TCLP Lead 50 – 150 (20)	MS/MSD ⁴	S & A
		Accuracy	No Detections exceeding the RL	Method Blank	A
		Accuracy	% Recoveries and RPD as follows: Cadmium 80-120 (20) Lead 80-120 (20) TCLP Cadmium 50-150 (20) TCLP Lead 50 – 150 (50)	Laboratory Control Sample	A
		Accuracy	RPD \leq 50% (or $\pm 2 \times$ RL if sample or duplicate is $< 5 \times$ RL)	QA split sample ⁵	S&A
		Sensitivity	RLs \leq Half the Action Level	RL Adequacy Check	A
		Field Completeness	100%	Data Completeness Check	S
		Analytical Completeness	100%	Data Completeness Check	A

Notes:

- 1) Reference number from QAPP Worksheet #21. Field SOPs are included in Appendix B.
- 2) Reference number from QAPP Worksheet #23. Laboratory SOPs are included in Appendix A.
- 3) Criteria from project laboratory. Limits are subject to change.
- 4) MS/MSDs will be performed on samples collected from the site. Note that the percent recovery criterion does not apply if the sample concentration exceeds four times the spike concentration.
- 5) QA split samples are collected at the discretion of the USEPA for analysis by an independent laboratory.

QAPP Worksheet #13
Secondary Data Criteria and Limitations Table

<i>Secondary Data</i>	<i>Data Source</i>	<i>Data Generator(s)</i>	<i>How Data Will Be Used</i>	<i>Limitations on Data Use</i>
Supplemental Investigation Results	Supplemental Investigation, CRA, Inc., 2009	CRA, Inc.	To locate and delineate the contaminated soil source areas to evaluate and implement possible Removal Actions	None: sampling and analytical methods and procedures were in accordance with the approved QAPP. The applicable data were validated and determined to be usable.

QAPP Worksheet #14 Summary of Project Tasks

Sampling Tasks:

Approximately 10 soil samples will be taken for confirmatory sampling.

Analysis Tasks:

PCBs: SW8082

Lead and Cadmium: SW 6010B

Dioxin: SW 8280A

TCLP Lead and Cadmium: SW1311/6010B

Quality Control Tasks:

The following QC samples will be analyzed: rinsate (field) blanks (if necessary), field duplicate, MS/MSDs, and QA split samples (if necessary).

Secondary Data:

None

Data Management Tasks:

Compile data into a summary table of analytical results (including corresponding action levels).

Documentation and Records:

Field data will be maintained in field books/field records and kept on file by CRA. The database will be stored electronically on CRA's network drive at the New Brighton, MN location.

Assessment/Audit Tasks:

A field audit may be conducted by the CRA QA manager during the sampling event. However, given the short duration of the sampling work, no field audits are anticipated to be needed. Laboratory audits (internal) will be conducted by the Test America Quality Manager (or designee) on an annual basis (minimum).

Data Review Tasks:

The laboratory will review all data prior to issuing an analytical report and verify analytical report completeness. CRA will prepare field data verification and validation (100% of data). CRA will prepare analytical data validation reports (100% of data). CRA will assess data usability as part of the Removal Plan. Data analysis (figures, tables, calculations, etc.) will be presented by CRA in the Construction Completion Report.

QAPP Worksheet #15

Reference Limits and Evaluation Table

Matrix: Soil
Analytical Group:
Concentration Level: Medium

<i>Method</i>	<i>SOP Number</i>	<i>Analytes</i>	<i>Action Level (mg/kg)</i>	<i>Source for Action Level⁽¹⁾</i>	<i>Project Reporting Limit Goal⁽²⁾ (mg/kg)</i>	<i>Laboratory Reporting Limit⁽³⁾ (mg/kg)</i>
8082	NC-GC-038	Aroclor 1016			0.033	0.033
PCB		Aroclor 1221			0.033	0.033
		Aroclor 1232			0.033	0.033
		Aroclor 1242			0.033	0.033
		Aroclor 1248			0.033	0.033
		Aroclor 1254			0.033	0.033
		Aroclor 1260			0.033	0.033
		Total PCB Aroclors	10	EPA		
8280A Dioxins	WS-ID-011, rev 4	2378-TCDD			0.5 ng/g	0.5 ng/g
		12378-PeCDD			1.25 ng/g	1.25 ng/g
		123478-HxCDD			1.25 ng/g	1.25 ng/g
		123678-HxCDD			1.25 ng/g	1.25 ng/g
		123789-HxCDD			1.25 ng/g	1.25 ng/g
		1234678-HpCDD			1.25 ng/g	1.25 ng/g
		OCDD			2.5 ng/g	2.5 ng/g
		Total TEQ Dioxin	1ug/kg	EPA		
Total Metals	NC-MT-012	Lead	400	EPA	0.3	0.3
	NC-MT-012	Cadmium	390	EPA	0.5	0.2
1311/6010	NC-MT-012, NC-OP-033	TCLP Lead	5 mg/L	TCLP Reg level		0.5 mg/L
TCLP Metals	NC-MT-012, NC-OP-033	TCLP Cadmium	1 mg/L	TCLP Reg level		0.1 mg/L

Notes:

*Reporting Limits will be reported as dry weight values.

1) The Action Level shown is the EPA or TCLP Regulatory Level.

2) Project Reporting Limit Goals are the Action Level.

3) Reporting Limits are equivalent to "practical quantitation limits". Method detection limits will generally be 2 to 10 times lower than reporting limits.

**QAPP Worksheet #16
Project Schedule/Timeline Table**

<i>Activities</i>	<i>Organization</i>	<i>Dates</i>		<i>Deliverable</i>	<i>Deliverable Due Date</i>
		<i>Anticipated Date(s) of Initiation</i>	<i>Anticipated Date of Completion</i>		
Prepare Draft Removal Plan and QAPP	CRA/PRP Group	Feb 2009	Feb 2009	Work Plan and QAPP Document	TBD
Approve Work Plan and QAPP	USEPA	TBD	TBD	Approval Letter	TBD
Field Work	CRA	TBD	TBD	Field Work	TBD
Laboratory Analysis	Test America	TBD	TBD	Analytical Report	TBD
Data Verification and Validation	CRA	TBD	TBD	Validation Reports (Include with Construction Completion Report)	TBD
Prepare Construction Completion Report	CRA/PRP Group	TBD	TBD	Construction Completion Report	TBD
Approve Construction Completion Report	USEPA	TBD	TBD	Approval Letter	TBD

QAPP Worksheet #17
(UFP-QAPP Manual Section 3.1.1)
Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):

Post-excavation confirmatory soil sampling will be conducted along excavation sidewalls (mid-height) at intervals not exceeding 25 feet, and over the excavation bottom at a frequency with grid nodes with spacing not exceeding 25 feet.

This sampling approach is commonly followed for soil removal projects and provides a sufficiently high frequency and density of sampling points to reasonably discern the soil contaminant levels that may be remaining in the existing soil.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer to map or Worksheet #18 for details]:

The sampling design will consist of approximately 20 soil samples collected for analysis of PCBs, Lead, Cadmium, TCLP Lead, and TCLP Cadmium. Sampling is a "one time" event and will be conducted in Spring 2009.

QAPP Worksheet #18
Sampling Locations and Methods/SOP Requirements Table

<i>Sampling Location/ID Number</i>	<i>Matrix</i>	<i>Depth (ft)</i>	<i>Analytical Group</i>	<i>Concentration Level</i>	<i>Number of Samples</i>	<i>Sampling SOP Reference</i>	<i>Rationale for Sampling Location</i>
TBD	Soil	TBD	PCBs	Medium	TBD	SOP-1	See Worksheet #17
TBD	Soil	TBD	TCLP Pb, Cd	Medium	TBD	SOP-1	See Worksheet #17
TBD	Soil	TBD	Total Pb, Cd	Medium	TBD	SOP-1	See Worksheet #17
TBD	Soil	TBD	Dioxins	Low	TBD	SOP-1	See Worksheet #17

QAPP Worksheet #19
Analytical SOP Requirements Table

<i>Matrix</i>	<i>Analytical Group</i>	<i>Concentration Level</i>	<i>Analytical and Preparation Method/SOP Reference</i>	<i>Sample Volume</i>	<i>Containers</i>	<i>Preservation Requirements</i>	<i>Maximum Holding Time (preparation/analysis)</i>
Soil	PCBs	Med	Modified SW-846 Method 8082 Test America SOP# (NC-GC-038 (Soils Preparation by NC-IP-010)	30 g of soil for extraction	4 oz glass Jar	4° ± 2°C	Extraction: 14 days from sample collection to extraction Analysis: 40 days from extraction to analysis
Soil	TCLP Metals	Med	SW-846 Method 1311/6010B Test America SOP# NC-MT-012-rev.0, NC-OP-033	1-2 g of soil	4 oz glass Jar	4° ± 2°C	180 days from sample collection to TCLP leachate; 180 days from leach to analysis
Soil	Total Metals	Med	SW-846 Method 6010 Test America SOP# NC-MT-012-rev.0	100 g of soil for digestion	4 oz glass Jar	4° ± 2°C	six months from collection to analysis
Soil	Dioxins	Low	SW-846 Method 8280A Test America SOP# WS-ID-011-rev 4	30 g of soil	4 oz glass Jar	4° ± 2°C	Extraction: 30 days from sample collection to extraction Analysis: 45 days from extraction to analysis

QAPP Worksheet #20
Field Quality Control Sample Summary Table

<i>Matrix</i>	<i>Analytical Group</i>	<i>Concentration Level</i>	<i>Analytical SOP Reference</i>	<i>No. of Sampling Locations</i>	<i>No. of Field Duplicate Pairs</i>	<i>No. of MS/MSDs</i>	<i>Total No. of Samples to Lab</i>
Soil	PCBs	Med	SOP# NC-GC-038	TBD	1:10	1:20	TBD
Soil	TCLP Pb., Cd	Med	SOP# NC-MT-012, NC-OP-033	TBD	1:10	1:20	TBD
Soil	Total Pb., Cd	Med	SOP# NC-MT-012	TBD	1:10	1:20	TBD
Soil	Dioxins	Low	SOP# WS-ID-011	TBD	1:10	1:20	TBD

Note:
 TBD - to be determined

QAPP Worksheet #21
Project Sampling SOP References Table

<i>Reference Number</i>	<i>Title, Revision Date and/or Number</i>	<i>Originating Organization</i>	<i>Modified for Project Work? (Y/N)</i>	<i>Comments</i>
SOP-1	Soil Sampling Procedures	CRA	Yes	Covers solid sampling method, field notes, and sample custody and shipment.

Note:

- 1) Sampling SOP is included in Appendix B.

QAPP Worksheet #22
Field Equipment Calibration, Maintenance, Testing, and Inspection Table

<i>Field Equipment</i>	<i>Calibration Activity</i>	<i>Maintenance Activity</i>	<i>Inspection Activity</i>	<i>Frequency</i>	<i>Acceptance Criteria</i>	<i>Corrective Action</i>	<i>Responsible Person</i>	<i>SOP Reference</i>
Sample Bottles	---	---	Note 1	Before each event	Certificate verifies clean	Replace with new bottles	Field Sampler	SOP-1

Note:

- 1) Review laboratory Certificates of Analysis to verify sample bottle cleanliness, and file this documentation.

QAPP Worksheet #23
Analytical SOP References Table

<i>Analytical SOP</i>	<i>Revision Number and Date</i>	<i>Definitive or Screening Data</i>	<i>Analytical Group</i>	<i>Instrument</i>	<i>Organization Performing Analysis</i>	<i>Modified for Project Work? (Y/N)</i>
SOP# NC-GC-038	Rev. 0 (6/08)	Definitive	PCBs	GC	Test America North Canton, OH	No
SOP# NC-MT-012, NC-OP-33	Rev. 0 (1/09), Rev 0 (3/08)	Definitive	TCLP Lead/Cadmium	ICP	Test America North Canton, OH	No
SOP# NC-MT-012	Rev. 1 (1/09)	Definitive	Total Lead/Cadmium	ICP/MS	Test America North Canton, OH	No
SOP# WS-ID-011	Rev. 4 (10/08)	Definitive	Dioxins	GC/MS	Test America West Sacramento, CA	No

Note:

- 1) Laboratory SOPs are included in Appendix A.

QAPP Worksheet #24
Analytical Instrument Calibration Table

<i>Instrument</i>	<i>Calibration Procedure</i>	<i>Frequency of Calibration</i>	<i>Acceptance Criteria</i>	<i>Corrective Action</i>	<i>Person Responsible for CA</i>	<i>SOP Reference¹</i>
GC	Refer to the laboratory SOP, section 10	Refer to the laboratory SOP, section 10	Refer to the laboratory SOP, section 10	Refer to the laboratory SOP, section 10	Laboratory Analyst	SOP# NC-GC-038
ICP	Refer to the laboratory SOP, section 10	Refer to the laboratory SOP, section 10	Refer to the laboratory SOP, section 10	Refer to the laboratory SOP, section 10	Laboratory Analyst	SOP# NC-MT-012
GC/MS	Refer to the laboratory SOP, section 10	Refer to the laboratory SOP, section 10	Refer to the laboratory SOP, section 10	Refer to the laboratory SOP, section 10	Laboratory Analyst	SOP# WS-ID-011

Note:

1) Laboratory SOPs are included in Appendix A.

QAPP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

<i>Instrument/ Equipment</i>	<i>Maintenance Activity</i>	<i>Testing Activity</i>	<i>Inspection Activity</i>	<i>Frequency</i>	<i>Acceptance Criteria</i>	<i>Corrective Action</i>	<i>Responsible Person</i>	<i>SOP Reference¹</i>
GC	Replace consumables, bake out column, recondition column.	Perform daily check.	Check connections, replace consumables, bake out column, recondition column, and perform leak test	See NC-GC-038, section 10	See NC-GC-038, section 10	Inspect system; correct problem and/or re-calibrate; re-analyzed all affected investigative samples.	Lab analyst	NC-GC-038
ICP	Replace consumables, stabilize ICP spec.	Perform daily check.	Check connections, replace consumables, stabilize ICP spec, and perform leak test	See NC-MT-012, section 10	See NC-MT-012, section 10	Inspect system; correct problem and/or re-calibrate; re-analyzed all affected investigative samples.	Lab analyst	NC-MT-012
GC/MS	Replace consumables, bake out column, recondition column.	Perform daily autotune.	Check connections, replace consumables, bake out column, recondition column, and perform leak test	See WS-ID-011, section 10	See WS-ID-011, section 10	Inspect system; correct problem and/or re-calibrate; re-analyzed all affected investigative samples.	Lab analyst	WS-ID-011

Note:

1) Laboratory SOPs are included in Appendix A.

QAPP Worksheet #26
Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Sampling Personnel (CRA)
Sample Packaging (Personnel/Organization): Sampling Personnel (CRA)
Coordination of Shipment (Personnel/Organization): Sampling Personnel or Lead Field Staff (CRA)
Type of Shipment/Carrier: Overnight Courier (Fed-Ex or UPS)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Management Personnel (Test America)
Sample Custody and Storage (Personnel/Organization): Sample Custodian or Analyst (Test America)
Sample Determinative Analysis (Personnel/Organization): Analyst (Test America)
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Typically no more than 1 day prior to shipments to laboratory
Total Sample Storage Before Analysis (No. of days from sample collection): See Worksheet #19
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodian (Test America)
Number of Days from Analysis: Not sooner than 30 days after reporting is complete

QAPP Worksheet #27
Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

See Field SOP-1

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

See the following laboratory SOPs/Quality Assurance Manual::

NC-QAM-001 2008 Rev 0, section 24, Figure 24-3 Internal Chain of Custody

NC-SC--005 Sample Management/receiving

Facility Addendum2007 Waste Handling

Sample Identification Procedures:

See Field SOP-1

Chain-of-Custody Procedures:

See Field SOP-1

Notes:

- 1) Field SOPs are included in Appendix B.
- 2) Laboratory SOPs are included in Appendix A.

QAPP Worksheet #28
QC Samples Table

<i>Matrix</i>	<i>Soil</i>				
<i>Analytical Group</i>	<i>PCBs</i>				
<i>Concentration Level</i>	<i>Med</i>				
<i>Sampling SOP (1)</i>	<i>1</i>				
<i>Analytical SOP (2)</i>	<i>SOP# NC-GC-038</i>				
<i>Field Sampling Firm</i>	<i>CRA</i>				
<i>Analytical Organization</i>	<i>Test America</i>				
<i>No. of Sample Locations</i>	<i>TBD</i>				
QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Method Blank	1 per Prep Batch ⁽⁴⁾	< RL	Note 3	Analyst	Accuracy
LCS	1 per Prep Batch	See Worksheet # 12	Note 3	Analyst	Accuracy
MS/MSD	1 per SDG ⁽⁵⁾	See Worksheet #12	Note 3	Analyst	Accuracy and Precision
Surrogates	Every Sample	See Worksheet #12	Note 3	Analyst	Accuracy
Internal Standards	Every Sample	Area Counts -50 to +100%	Note 6	Analyst	Accuracy and Precision
Initial and Continuing Calibration	Note 6	Note 6	Note 6	Analyst	Accuracy
Field Duplicates/rinse blanks	10% for project ⁽⁷⁾	Note 8	Note 8	Note 8	Precision

<i>Matrix</i>	<i>Soil</i>
<i>Analytical Group</i>	<i>Total/TCLP Metals</i>
<i>Concentration Level</i>	<i>Med</i>
<i>Sampling SOP (1)</i>	<i>1</i>
<i>Analytical SOP (2)</i>	<i>SOP# NC-MT-012</i>
<i>Field Sampling Firm</i>	<i>CRA</i>
<i>Analytical Organization</i>	<i>Test America</i>
<i>No. of Sample Locations</i>	<i>TBD</i>

<i>QC Sample:</i>	<i>Frequency/Number</i>	<i>Method/SOP QC Acceptance Limits</i>	<i>Corrective Action</i>	<i>Person(s) Responsible for Corrective Action</i>	<i>Data Quality Indicator (DQI)</i>
Method Blank	1 per Prep Batch ⁽⁴⁾	< RL	Note 3	Analyst	Accuracy
LCS	1 per Prep Batch	See Worksheet # 12	Note 3	Analyst	Accuracy
MS/MSD	1 per SDG ⁽⁵⁾	See Worksheet #12	Note 3	Analyst	Accuracy and Precision
Initial and Continuing Calibration	Note 6	Note 6	Note 6	Analyst	Accuracy
Field Duplicates	10% for project ⁽⁷⁾	Note 8	Note 8	Note 8	Precision

<i>Matrix</i>	<i>Soil</i>
<i>Analytical Group</i>	<i>Dioxins</i>
<i>Concentration Level</i>	<i>Low</i>
<i>Sampling SOP (1)</i>	<i>1</i>
<i>Analytical SOP (2)</i>	<i>SOP# WS-ID-011</i>
<i>Field Sampling Firm</i>	<i>CRA</i>
<i>Analytical Organization</i>	<i>Test America</i>
<i>No. of Sample Locations</i>	<i>TBD</i>

<i>QC Sample:</i>	<i>Frequency/Number</i>	<i>Method/SOP QC Acceptance Limits</i>	<i>Corrective Action</i>	<i>Person(s) Responsible for Corrective Action</i>	<i>Data Quality Indicator (DQI)</i>
Method Blank	1 per Prep Batch ⁽⁴⁾	< RL	Note 3	Analyst	Accuracy
LCS	1 per PrepBatch	See Worksheet # 12	Note 3	Analyst	Accuracy
MS/MSD	1 per SDG ⁽⁵⁾	See Worksheet #12	Note 3	Analyst	Accuracy and Precision
Internal Standards	Every Sample	25-150%	Note 6	Analyst	Accuracy and Precision
Initial and Continuing Calibration	Note 6	Note 6	Note 6	Analyst	Accuracy
Field Duplicates/rinse blanks	10% for project ⁽⁷⁾	Note 8	Note 8	Note 8	Precision

Notes:

- 1) Field SOPs are included in Appendix B.
- 2) Laboratory SOPs are included in Appendix A.
- 3) Refer to Test America SOP# WS-IDP-011, section 9.
- 4) A preparation batch is defined as any group of samples of the same matrix that are prepared together.
- 5) A Sample Delivery Group (SDG) will consist of up to 10 samples of the same matrix that are analyzed and reported together.
- 6) Refer to Test America SOP# WS-IDP-011, section 7
- 7) Field duplicates and equipment rinse blanks will be collected at a rate of 10% for the project.
- 8) There are no Method/SOP acceptance limits or corrective action for these QC samples.

QAPP Worksheet #29 **Project Documents and Records Table**

<i>Sample Collection Documents and Records</i>	<i>Off-site Analysis Documents and Records</i>	<i>Data Assessment Documents and Records</i>
Field Notes	Sample Receipt, Custody, and Tracking Records	Laboratory Audit Forms
Chain-of-Custody	Standard Traceability Logs	Data Verification Forms
Air Bills	Equipment Calibration Logs	Data Validation Reports
Custody Seals	Sample Prep Logs	Corrective Action Forms
Telephone Logs and E-mail	Run Logs	Telephone Logs and E-mail
Corrective Action Forms	Equipment Maintenance, Testing, and Inspection Logs	
Certificates of Analysis (bottle cleanliness)	Corrective Action Forms	
	Reported Field Sample Results	
	Reported Results for Standards, QC Checks, and QC Samples	
	Instrument Printouts (raw data) for Field Samples, Standards, QC Checks, and QC Samples	
	Telephone Logs and E-mail	
	Extraction/Clean-up Records	
	Raw Data (electronically stored)	

Employee Orientation and Training
Records Information Management
Preparation and Management of Standard Operating Procedures
Data Validation Response
Sample Receiving and Sample Control

CORP-QA-0013NC
NC-QA-019
NC-QA-0027
QA-020
NC-SC-005

Notes:

- 1) CRA will retain sample collection and data assessment documents and records for a minimum period of five years.
- 2) Test America will retain Analytical documents and records for a minimum period of 5 years, after which they will transfer files to CRA, who will retain them for a minimum period of 10 years.
- 3) Laboratory SOPs for documentation and records include:

QAPP Worksheet #29
Project Documents and Records Table (continued)

Data Reporting Formats:

Field data recording procedures for soil sampling are specified in Field SOP-1 in Appendix B. Procedures for sample bottle labeling are specified in Field SOP -1 in Appendix B. Procedures for filling out field chain-of-custody forms are specified in Field SOP-1 in Appendix B. Example chain-of-custody form, sample label and custody seal are included at the end of Field SOP-1 in Appendix B.

The laboratory data package contents will include, at a minimum:

1. Cover Page
2. Case Narrative
3. Corrective Action Form (if applicable)
4. Sample Results
5. Method QC Results (method blank, LCS, MS/MSD, internal stds (dioxins), surrogates (PCBs))
6. Chain-of-Custody (field)
7. Sample Receipt Log
8. Shipping Label (if applicable)

Test America procedures for data package preparation and review are described in Laboratory SOP# NC-AD-001 and NC-AD-002 (Appendix A). Test America procedures for recording data in notebooks/logbooks are described in Laboratory SOP# NC-QAM-001 (Appendix A).

Data Handling and Management:

Test America's data management system is described Laboratory SOP# NC-QA-019 (Appendix A). This SOP describes the software that is used to acquire analytical results and generate deliverables, data management, procedures for reducing data entry errors, and procedures for correcting errors. Calculations that are utilized by Test America are performed in accordance with the method SOPs. If manual integrations are performed on any samples, QC samples, or calibration standards, Test America will flag such data in their analytical report and will also provide chromatograms from before and after the manual integration, along with the reason for the manual integration.

QAPP Worksheet #29
Project Documents and Records Table (continued)

Prior to sending the report, the Test America Project Manager will verify that analytical reports are complete and technically accurate. Test America sends analytical results to the CRA QA Manager both as a hard copy of the data package (by mail) and also an electronic data deliverable (EDD) by e-mail. The EDD is in a format that facilitates direct entry into data tables to minimize data handling errors, such that no data entry or data entry forms are required. CRA does not perform any data transformation or calculations that would alter the data values received from Test America.

The hard copy of the deliverable is then forwarded by CRA's data validator. The data validator will also verify analytical report completeness as part of the data validation process. The data validator will validate the analytical data as discussed in Worksheet #35. If during the validation process it is realized that corrections need to be made to the deliverable, they will be made at that time, and a corrected deliverable and EDD will be resent by Test America. The EDD is then used to create/update the tables (Excel) that ultimately appear in the Construction Completion Report. Any data qualifiers that were assigned to the data during the data validation process are manually added into the Excel tables. These tables, with qualifiers, will ultimately appear in the Construction Completion Report. A second individual from within CRA will proof the manually added data qualifiers and the analytical results from the hard copy data package against the data tables that will appear in the Construction Completion Report to check for any data entry errors.

Data Tracking and Control:

The CRA QA Manager tracks the reporting of analytical data and coordinates analytical data validation. The CRA QA Manager also tracks and manages the updating and storage of the data tables (Excel) described above. These tables are stored on a network drive at CRA's New Brighton, Minnesota office. This network drive is backed up nightly, with additional backup tapes from other increasing time intervals also being concurrently stored at any given time, which minimizes the potential of losing the most current versions of these databases. Data stored electronically by CRA will be retrievable for at least five years.

The Test America Project Manager will verify that laboratory documents and records (as listed at the beginning of this worksheet) are properly filed. Test America will archive the final report and associated raw data for all analyses for a minimum period of 5 years (either in the file storage area of the laboratory or at their subcontracted storage facility). After 5 years, Test America may transfer these files to CRA, who will retain these files for a minimum period of five years. Test America's procedures for archiving final analytical reports and associated raw data are described in SOP# NC-QA-019 (Appendix A).

Electronic data storage is discussed in Test America SOP# NC-QA-019 (Appendix A).

QAPP Worksheet #30
Analytical Services Table

<i>Matrix</i>	<i>Analytical Group</i>	<i>Concentration Level</i>	<i>Sample Locations/ID Numbers</i>	<i>Analytical SOP</i>	<i>Data Package Turnaround Time</i>	<i>Laboratory/ Organization</i>
Soil	PCBs	Medium	Confirmatory soil sample locations	NC-GC-038	2 weeks	Test America North Canton, OH
Soil	Total and TCLP Metals	Medium	Confirmatory soil sample locations	NC-OP-033 NC-MT-012	2 weeks	Test America North Canton, OH

QAPP Worksheet #31
Planned Project Assessments Table

<i>Assessment Type</i>	<i>Frequency</i>	<i>Internal or External</i>	<i>Organization Performing Assessment</i>	<i>Person(s) Responsible for Performing Assessment and Identifying Corrective Actions</i>	<i>Person(s) Responsible for Implementing Corrective Actions</i>	<i>Person(s) Responsible for Monitoring Effectiveness of Corrective Actions</i>
Field Sampling Technical System Audit	As necessary	Internal	CRA	CRA QA Manager (or Senior QA personnel)	CRA Lead Sampling Staff	CRA QA Manager
Laboratory Technical Performance Audit	Annual (minimum)	Internal	Test America	Test America Quality Manager (or designee)	Test America Laboratory Supervisor	Test America Quality Manager

Notes:

1) Test America is externally audited to maintain certifications.

- 2) Test America will not be routinely audited by the USEPA or The Group; however, the USEPA reserves the right to audit the laboratory, and if such audit reveals significant issues, The Group will be asked to participate in the audit process.

QAPP Worksheet #32
Assessment Findings and Corrective Action Responses

<i>Assessment Type</i>	<i>Nature of Deficiencies Documentation</i>	<i>Individual(s) Notified of Findings</i>	<i>Timeframe of Notification</i>	<i>Nature of Corrective Action Response Documentation</i>	<i>Individual(s) Receiving Corrective Action Response</i>	<i>Timeframe for Beginning Implementation of Response</i>
Field Sampling Technical System Audit	Written Audit Report	CRA QA Manager and CRA Project Manager ⁽¹⁾	ASAP and within 1 business day	E-mail or Letter	CRA Lead Field Staff	1 business day
Laboratory Technical Performance Audit	Written Audit Report	Test America Project Manager and General Manager ⁽²⁾	ASAP and within 1 business day	Corrective Action Form	Test America Laboratory Supervisor and General Manager	1 business day

Notes:

- 1) CRA Project Manager will notify The Group within two business days, who will then notify USEPA Project Manager within two business days.
- 2) Laboratory Project Manager will notify the CRA QA Manager within one business day. The CRA QA Manager will then notify the CRA Project Manager and The Group within two business days, who will then notify USEPA Project Manager within two business days.

QAPP Worksheet #33
QA Management Reports Table

<i>Type of Report</i>	<i>Frequency</i>	<i>Projected Delivery Date(s)</i>	<i>Person(s) Responsible for Report Preparation</i>	<i>Report Recipient(s)</i>
Laboratory Technical Performance Audit	Annual (at a minimum)	Ongoing	Test America Quality Manager	Available Upon Request
Data Verification and Validation Reports	Each Sampling Event	Submitted with Construction Completion Report (see below)	CRA QA Manager	Submitted with Construction Completion Report
Data Usability Assessment	Part of Construction Completion Report	Submitted with Construction Completion Report (see below)	CRA QA Manager	Submitted with Construction Completion Report
Construction Completion Report	(Final Report)	2009	CRA Project Manager	The Group, USEPA

QAPP Worksheet #34
Verification (Step I) Process Table

<i>Verification Input</i>	<i>Description</i>	<i>Internal/ External</i>	<i>Responsible for Verification (Name, Organization)</i>
Field Documentation	Field Documentation will be reviewed to verify that required documentation was completed by the field sampler. For this verification step, the field sampler will provide copies of field notes, chain-of-custody, Certificate of Analysis (bottle cleanliness), and any information regarding sampling deviation or corrective action. Verify that all required samples were collected and that all required analyses/analytes were requested.	Internal	CRA QA Manager
Field Audit and Corrective Action Documentation	Verify that field audit and corrective action documentation (if any) are completed and properly filed.	Internal	CRA Project Manager
Laboratory Documentation	Verify that all laboratory documentation (see Worksheet #29) is properly filed in accordance with laboratory SOPs.	Internal	Test America Project Manager
Analytical Report	Verify that analytical reports are complete and technically accurate prior to mailing to CRA (see Note 1).	Internal	Test America Project Manager
Laboratory Audit and Corrective Action Forms	Verify that laboratory audit forms and corrective action forms (if any) are completed and properly filed.	Internal	Test America QA Manager

Note:

1) Verification of analytical report completeness will also be conducted externally by CRA Data Validator as part of Data Validation (see Worksheet #35).

QAPP Worksheet #35
Validation (Steps IIa and IIb) Process Table

<i>Step IIa/IIb</i>	<i>Validation Input</i>	<i>Description</i>	<i>Responsible for Validation</i>
IIa / IIb	Field Documentation	Review the items listed in field SOP/audit form to ensure that QAPP-specified sampling and documentation procedures were followed.	CRA QA Field Staff/PM
IIa / IIb	Analytical Data	Review the items listed in Worksheet #29 to ensure that QAPP-specified requirements were met and to determine any required data qualification based on QAPP performance criteria (see Worksheet #12).	Data Validator

Note:

1) Validation of field and analytical data will be performed on 100% of the data collected in the project.

QAPP Worksheet #36
Validation (Steps IIa and IIb) Summary Table

<i>Step IIa/IIb</i>	<i>Matrix</i>	<i>Analytical Group</i>	<i>Concentration Level</i>	<i>Validation Criteria</i>	<i>Data Validator</i>
IIa / IIb	Soil	PCBs	Med	Note 1	Grant Anderson
IIa / IIb	Soil	Total, TCLP Metals	Med	Note 1	Grant Anderson
IIa / IIb	Soil	Dioxins	Low	Note 1	Grant Anderson

Note:

- 1) Compliance with methods and procedures (Step IIa) will be determined by comparison with the QAPP requirements. Compliance with method performance criteria (Step IIb) will be determined by comparison with QAPP-specified performance criteria (Worksheets 12, 15, and 20). National Functional Guidelines for Organic Data Review (USEPA, October 1999) and Inorganic Data Review (USEPA, October 2004) will be used as guidance where QAPP requirements are not specific.

QAPP Worksheet #37 Usability Assessment

Summarize the usability assessment process:

Final determination of data usability will be made as part of the Construction Completion Report, wherein the quality of the sampling data used in decision-making will be evaluated as further described below.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

Data validation reports (field and analytical) will be reviewed for any deviations from QAPP-specified procedures. If any deviations were noted, the potential measurement error will be considered. Completeness, accuracy, precision, and sensitivity, will all have been evaluated through data validation of each event's data. These items will be summarized and discussed relative to potential measurement error.

Identify the personnel responsible for performing the usability assessment:

Data usability determination will be conducted by the CRA PM. No statistical evaluations are envisioned; however; if any such evaluations are performed (e.g. outlier determination), the CRA PM will seek assistance from CRA personnel having statistics expertise.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

Data usability evaluation, discussion, and conclusions will be presented in the Construction Completion Report. Tables summarizing QC results for data quality from project sampling events will be presented and evaluated, including these tables: method blanks, laboratory control samples, matrix spikes and MS/MSD precision, and field duplicates. Presenting these results in tabular form will provide for project-wide evaluation of data quality trends and potential bias. The data usability discussion and conclusion will indicate whether project data met the data quality objectives (DQOs) specified in this QAPP.

In addition to validation, the data will be examined to determine if individual or batch QC exceedances affect project decisions. In the same manner, trends in QC results and potential impacts upon data usability will be evaluated and discussed as part of the data assessment.

QC sample results (e.g., MS/MSD and field duplicate relative percent differences [RPDs]) will be evaluated to determine if observed sample heterogeneity significantly impacts the ability to reconcile the data to the DQOs. If RPD results exceed the acceptance limits listed given in Worksheet #12, and sample heterogeneity is determined to be a significant concern, additional sampling and/or other means of reducing sample variability may need to be considered in order to ensure project DQOs are met.

APPENDIX A
LABORATORY SOPs

APPENDIX A


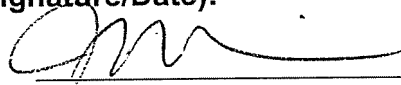


TABLE OF CONTENTS LABORATORY STANDARD OPERATING PROCEDURES

Laboratory Standard Operating Procedures

<i>Title</i>	<i>SOP</i>
Analysis for PCDDs/PCDFs for Low Resolution GC/MS analysis	WS-ID-0011
Extraction for PCDDs/PCDFs for Low Resolution GC/MS analysis	WS-IDP-011
ICP-AES, Spectrometric Method for Trace Element Analyses	NC-MT-012
Acid Digestion of Aqueous Samples	NC-IP-011
Acid Digestion of Soil Samples	NC-IP-010
Gas Chromatographic Analysis Based on Methods 8000B, 8021B, 8081A, 8082, 8151A, 8015B, 615	NC-GC-038
Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure SW846 Method 1311, SW-846 Method 1312	NC-OP-033
Employee Orientation and Training	CORP-QA-0013NC
Records Information Management	NC-QA-019
Preparation and Management of Standard Operating Procedures	NC-QA-0027
Data Validation Response	QA-020
Sample Receiving and Sample Control	NC-SC-005
Quality Assurance Manual	NC-QAM-001
Facility Addendum & Contingency Plan	Facility Addendum & Contingency Plan
Project Management	NC-AD-001
Central Reporting	NC-AD-002

**Title: Analysis of Polychlorinated Dibenzo-p-dioxins and
Dibenzofurans by Methods 8280A and 8280B**

[Methods 8280A and 8280B]

Approvals (Signature/Date):			
	10/01/08		9/30/08
Douglas Weir	Date	Joe Schairer	Date
Technical Manager		Health & Safety Manager / Coordinator	
	10/1/08		10/1/08
Pamela Schemmer	Date	Karla Buechler	Date
Quality Assurance Manager		Laboratory Director	

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1. SCOPE AND APPLICATION

- 1.1. This procedure is appropriate for the determination of tetra-, penta-, hexa-, hepta-, and octachlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) in water, soil, sediment, fly ash, and chemical waste samples per methods 8280A and 8280B. The analytical method requires the use of high resolution gas chromatography and low resolution mass spectrometry on sample extracts that have been subjected to specified cleanup procedures.
- 1.2. The calibration range is dependent on the compound and sample size. The sample size varies by sample matrix. The upper limit of the calibration range for each compound is 20 times the Lower Calibration Limit (LCL).
- 1.3. Typically, the lowest calibration standard is used as the reporting and flagging limit. See Table 1 for reporting limit/LCL.
- 1.4. Sample preparation is by Method 8280A or 8280B, per SOP WS-IDP-0011.

2. SUMMARY OF METHOD

- 2.1. A specified amount of water, soil, sediment, fly ash or chemical waste samples is spiked with the internal standard solution. Soil, sediment, fly ash or chemical waste samples are extracted with toluene in a combination of Soxhlet extractor (or equivalent Soxtherm or Soxtech system) with a Dean Stark water separator (SDS). Aqueous samples are filtered prior to extraction, and the glass fiber (or cellulose) filter paper extracted as a solid. The filtered aqueous fraction is extracted with methylene chloride using a separatory funnel. The extract is subjected to clean up procedures prior to analysis. Sample preparation and cleanup is addressed in SOP WS-IDP-0011.
- 2.2. A 2 µL aliquot is injected into a fused silica capillary column in a gas chromatograph (GC) interfaced to a mass spectrometer (MS). The mass spectrometer can either be low or high resolution models. Identification of PCDDs/PCDFs is based on the detection of the ions specified in Table 2a for the low resolution mass spectrometer (LRMS) and in Table 2b for the high resolution mass spectrometer (HRMS), and the measurement of the appropriate relative abundance (ratio) of two characteristic ions in the molecular ion cluster. The PCDDs/PCDFs are quantitated by comparing the MS response of the detected analyte relative to the MS response of the appropriate internal standard.

3. DEFINITIONS

- 3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

- 3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.
- 3.3. Internal Standards: Internal standards are isotopically labeled analogs which are present at the same concentration in every blank, field sample, quality control sample, and calibration solution. The internal standards are added to the sample before extraction and are used to measure the concentrations of the analytes.
- 3.4. Recovery Standard: Recovery standard is added to every blank, quality control sample, and sample extract aliquot prior to analysis. Recovery standards are used to measure the recovery of the internal standards.
- 3.5. Cleanup Standard: Cleanup standard is added to every sample, blank, quality control sample, and concentration calibration solution. It is added to the samples after extraction but prior to cleanup, to permit evaluation of the cleanup process.
- 3.6. Signal-to-noise: The ratio of analyte signal to random background signal.
- 3.7. Estimated Detection Limit (EDL): The concentration of an analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. The EDL is calculated for each 2,3,7,8-substituted isomer for which the response of the quantitation and confirmation ions is less than 2.5 times the background level.
- 3.8. Window Defining Mix: A solution that contains the first and last eluting isomer of each homologue and is used to verify that the switching times between the descriptors have been properly set.
- 3.9. Homologous Series: A series of organic compounds in which each successive member has one more chlorine atom than the preceding member.
- 3.10. Isomer: Chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are structural isomers.
- 3.11. Congener: Any member of a particular homologous series, for example, pentachlorinated dibenzofurans.

4. INTERFERENCES

- 4.1. Sample components may interfere with the detection and measurement of PCDDs/PCDFs. To reduce interferences, the extract is cleaned up by column chromatographic procedures.
- 4.2. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines which may cause misinterpretation of

chromatographic data. All of these materials shall be demonstrated to be free from interferences under the conditions of analysis by running laboratory method blanks. Analysts must avoid using PVC gloves due to the possibility of contamination.

- 4.3. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all glass systems may be necessary.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

- 5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.
- 5.1.2. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.1.3. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.
- 5.1.4. The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

5.2. Primary Material Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Gas Chromatograph/Mass Spectrometer/Data System (GC/MS/DS)

- 6.1.1. The GC must be capable of temperature programming and equipped with all required accessories, such as syringes, gases, and a capillary column. The GC injection port is designed for capillary columns; a splitless technique is recommended.
- 6.1.2. The mass spectral data obtained using a low resolution instrument utilizes 70 volts (nominal) electron energy in the electron impact mode. The system is capable of selected ion monitoring (SIM) for at least 18 ions simultaneously, with a cycle time of 1 second or less. Minimum integration time for SIM is 25 millisecond per m/z. The integration time used to analyze samples is identical to the time used to analyze the calibration solutions and QC samples. Total data acquisition time per cycle (18 ions) must not exceed 1 second.
- 6.1.3. The mass spectral data obtained using a high resolution instrument uses appropriate electron energy in the electron impact mode to obtain a calibration with required sensitivity for the CC1 level compounds (Section 10.4.3.2) and so the CC5 level does not saturate. The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less.
- 6.1.4. An interfaced data system is present to acquire, store, reduce, and output mass spectral data.

6.2. GC Columns

- 6.2.1. Fused silica capillary columns are required. The columns shall demonstrate the required separation of all 2,3,7,8-specific isomers whether a dual column or a single column is chosen. Column operating conditions shall be evaluated at the beginning and end of each analytical sequence during which samples or calibration solutions are analyzed.
- 6.2.2. Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60 m DB-5 column. In order to determine the concentration of the individual 2,3,7,8-substituted isomers, if the toxicity equivalence is greater than 0.7 ppb (solids), 7 ppt (aqueous), or 7 ppb (chemical waste), then the sample extract may need reanalysis on a 60 m DB-225 (or equivalent) GC column depending upon client requirements and site history.

7. REAGENTS AND STANDARDS

7.1. Solvents: High purity, distilled-in-glass: tetradecane.

7.2. Calibration Solutions

- 7.2.1. All PCDD/PCDF calibration, internal standard, clean-up recovery standards, and spike working solutions are stable for at least one year from preparation. After one year, solutions may be reverified. The reverification solution may be used for an additional year or until there is evidence of compound degradation or concentration. The reverification must be performed using an unexpired, not previously verified solution from a second lot or second vendor. Stock standards are stable for 10 years. For more details on standard expiration and reverification, see SOP WS-QA-0017.
- 7.2.2. Five tetradecane solutions (CC1-CC5) containing at least 10 unlabeled and at least 7 carbon-labeled PCDDs/PCDFs at known concentrations are used to calibrate the instrument. See Table 3 for calibration concentrations. One of these five solutions (CC3) is used as the continuing calibration solution and contains at least 7 additional unlabeled 2,3,7,8-substituted isomers that are commercially supplied. The concentration ranges are homologue-dependent with the lowest concentrations associated with tetrachlorinated dioxins and furans (0.1-2.0 ng/μL), and higher concentrations associated with the penta-through octachlorinated homologues (0.5 ng/μL-10.0 ng/μL). The CC3 is used as the continuing calibration and the CC1 is used to demonstrate instrument sensitivity.
- 7.2.3. Store standard solutions at $\leq 6^{\circ}\text{C}$.

- 7.3. The window defining mix is a solution obtained by the laboratory through a commercial vendor. This solution contains the first and last eluting isomer of each homologue (See Table 4) and is used to verify that the switching times between the descriptors have been properly set. The window defining mix need not contain any of the labeled internal or recovery standards, as no quantitative measurements are based on this mixture.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. All samples should be stored at $4^{\circ} \pm 2^{\circ}\text{C}$ in the dark and extracted within 30 days of collection and completely analyzed within 45 days of extraction. Whenever samples are analyzed after the holding time expiration date, the results should be flagged appropriately.
- 8.2. Protect samples from light at the time of receipt until extraction to minimize the potential for photodecomposition.
- 8.3. For Method 8280B, store all samples and extracts at $\leq 6^{\circ}\text{C}$ in the dark.

NOTE: The holding times listed above are recommendations, as noted by the method. PCDDs and PCDFs are very stable in a variety of matrices, and holding times under the conditions listed above may be as high as a year for certain matrices. Sample extracts, however, should always be analyzed within 45 days of extraction.

9. QUALITY CONTROL

- 9.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented in a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when internal standard recoveries are outside control limits. Re-extraction of the blank, other batch QC and the affected samples are required when the method blank is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria.

- 9.1.1. Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than $\frac{1}{2}$ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for some matrices including wipes are generally not possible because the entire sample is consumed in the initial extraction. Re-processing of the archive is the only available check of the process in this case.

- 9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented in a Non-Conformance memo, then implemented when the recoveries of any spiked analyte is outside control limits. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria. The LCS and MS/MSD levels are in Table 5.
- 9.3. The assessment of matrix effects on method performance, as required by NELAP, is met, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance can be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair is extracted at the client's request only. An exception to this rule is a batch containing South Carolina samples. These batches must have an MS/MSD prepared. The MS/MSD are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recovery of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.
- 9.3.1. Matrix Spike (MS): A sample that is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined and used to estimate the effect of the sample matrix upon the analytical methodology.
- 9.3.2. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.
- 9.4. Duplicates
- 9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or

representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.1.2. Certain programs, such as DoD QSM, may have more stringent requirements for the RPD criterion. In the case of the DoD QSM, the RPD must be less than or equal to 20%.

9.5. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

10. CALIBRATION

10.1. Mass Calibration

10.1.1. LRMS: Mass calibration of the mass spectrometer is recommended prior to analyzing the continuing calibration solution or samples each day. Tune the instrument for greater sensitivity in the high mass range to achieve better response to the later eluting compounds.

10.1.2. HRMS: The recommended mass spectrometer tuning conditions are based on the groups of monitored ions using a perfluorokerosene (PFK) molecular leak. Tune the instrument to meet the minimum required resolving power of 2,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF).

10.2. Window Defining Mix (WDM)

10.2.1. Prior to the calibration of the GC/MS system, it is necessary to establish the appropriate switching times for the SIM descriptors and to verify the chromatographic resolution. The switching times are determined by the analysis of the window defining mix that contains the first and last eluting isomer in each homologue. (See Table 4)

10.2.2. The window defining mix is analyzed at the initiation of an analytical sequence prior to analyzing samples.

10.3. Chromatographic Resolution

For analyses on a DB-5 (or equivalent) GC column, the chromatographic resolution is evaluated by the analysis of the CC3 standard during both the initial and continuing calibration procedures.

Note: the continuing standard concentration may be varied with a CC2 or CC4 standard.

GC Resolution for DB-5 or equivalent column. The chromatographic peak separation between ¹³C-2,3,7,8-TCDD peak and ¹³C-1,2,3,4-TCDD isomers shall be resolved with a valley of < 25%, in all calibration standards.

$$\text{Valley} = \frac{X}{Y} \times 100$$

Where:

X = the height from the baseline to the bottom of the valley between adjacent peaks.

Y = the peak height of the shorter of the two isomers.

- 10.3.1. For the continuing calibration verification, the chromatographic peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD in the CC3 solution shall be resolved with a valley of < 50%, calculated in a similar fashion as above. The resolution criteria must be evaluated using measurements made on the SICP for the appropriate ions for each isomer. Measurements are not made from total ion current profiles.
- 10.3.2. The relative ion abundance criteria for PCDDs/PCDFs listed in Table 2, must be met for all PCDD/PCDF peaks, including the labeled internal and recovery standards, in all solutions. The lower and upper limits of the ion abundance ratios represent a $\pm 15\%$ window around the theoretical abundance ratio for each pair of selected ions. The ³⁷Cl₄-2,3,7,8-TCDD cleanup standard is only monitored by the ³⁷Cl ion, thus the ion abundance ratio criterion does not apply to this compound.
- 10.3.3. MS Sensitivity: For all calibration solutions including the CC1 solution, the signal-to-noise ratio (S/N) must be greater than 2.5 for the unlabeled PCDD/PCDF ions and greater than 10 for the internal standard and recovery standard ions.

10.4. Five-Point Initial Calibration

- 10.4.1. The five calibration solutions outlined in Table 3 must be analyzed prior to any sample analyses.
- 10.4.2. Analyze a 2 μ L aliquot of each of the five concentration calibration solutions. The identical GC/MS/DS conditions used for the WDM or CPSM solution

must also be used for the concentration calibration solutions.

- 10.4.3. The laboratory must not proceed with the sample analyses until an acceptable initial calibration is determined and documented according to the following criteria:

10.4.3.1. The relative ion abundance criteria listed for PCDDs/PCDFs in Table 2 must be met using areas to calculate the ratios.

10.4.3.2. MS Sensitivity: The signal-to-noise (S/N) ratio must be greater than 2.5 for the unlabeled PCDD/PCDF ions and greater than 10 for the internal standard and recovery standard ions.

10.5. Relative Response Factors

- 10.5.1. Calculate the relative response factors (RRFs) for the 17 unlabeled target analytes relative to their appropriate internal standards (RRF_n) according to the formula below for each calibration standard.

$$RRF_n = \frac{(A_{n1} + A_{n2}) \times Q_{is}}{(A_{is1} + A_{is2}) \times Q_n}$$

Where:

A_{n1}, A_{n2} = integrated areas of the two quantitation ions of the isomer of interest (Table 2).

A_{is1}, A_{is2} = integrated areas of the two quantitation ions of the appropriate internal standard (Table 2).

RRF_n = the response factor of the quantitation ions of the isomer of interest relative to that of the appropriate internal standard.

Q_n = quantity of unlabeled PCDD/PCDF analyte injected (ng).

Q_{is} = quantity of appropriate internal standard injected (ng).

- 10.5.2. Calculate the RRF_s for the five labeled internal standards and the cleanup standard relative to the appropriate recovery standard (RRF_{is}) in each calibration standard according to the formula below for each calibration standard.

$$RRF_{is} = \frac{(A_{is1} + A_{is2}) \times Q_{rs}}{(A_{rs1} + A_{rs2}) \times Q_{is}}$$

Where:

A_{is1}, A_{is2}, Q_{is} are defined above, and

A_{rs1}, A_{rs2} = integrated areas of the two quantitation ions of the appropriate recovery standard (Table 2).

RRF_{is} = the response factor of the internal standard relative to that of the appropriate internal standard.

Q_{rs} = quantity of appropriate recovery standard injected (ng).

10.5.3. There is only one quantitation ion for the ^{37}Cl cleanup standard. Calculate the relative response factor as described for RRF_{is} , using one area for the cleanup standard and the sum of the areas of the ions from the recovery standard.

10.5.4. The RRF_n and the RRF_{is} are dimensionless quantities; therefore, the units used to express the Q_n , Q_{is} , and Q_{rs} must be the same.

10.5.5. Calculate the relative response factors for the native PCDDs/PCDFs relative to the recovery standards (RRF_{rs}):

$$RRF_{rs} = RRF_n \times RRF_{is}$$

This is used when the sample is diluted to the extent that the MS response of the internal standard is less than 10:1 signal to noise due to matrix impacts and a re-extraction would not improve results. (See Section 12.4) The RRF_{rs} method of quantitation is only used when the sample is diluted to the extent that the S/N ratio for the internal standard is less than 10:1.

10.5.6. Response Factor Criteria

Calculate the mean RRF and percent relative standard deviation (%RSD) of the five RRFs for the unlabeled PCDDs/PCDFs (CC1-CC5) and labeled internal standards present in all five concentration calibration solutions.

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean RRF}} \times 100\%$$

The %RSD of the RRFs for the unlabeled PCDDs/PCDFs (CC1-CC5) and the internal standards must not exceed 15 percent for method 8280A, and 20 percent for method 8280B.

10.5.7. Initial calibration verification standard (ICV): When available, a second source calibration standard is analyzed with the initial calibration curve. Each compound of the ICV must be within $\pm 30\%$ of its expected value for method 8280A, and $\pm 20\%$ of its expected value for method 8280B.

10.6. Continuing Calibration (i.e. Daily Standard)

10.6.1. Inject a 2 μL aliquot of the continuing CC3 calibration solution. The continuing calibration solution must be analyzed at the beginning of each 12-hour period to evaluate chromatographic resolution and calculate the RRF values used in quantitation. Use the equations in Section 10.5 to calculate the RRF values for the continuing calibration.

- 10.6.2. The identical GC/MS/DS conditions used for the WDM and the initial calibration solutions must also be used for the continuing calibration solution.
- 10.6.3. GC Column Resolution Criteria. The chromatographic resolution on the DB-5 (or equivalent) column must meet the QC criteria in Section 10.3.
- 10.6.4. Ion Abundance Criteria. The relative ion abundances listed in Table 2 must be met for all PCDD/PCDF peaks, including the labeled internal and recovery standards.
- 10.6.5. Instrument Sensitivity Criteria. For the CC3 solution, the S/N ratio must be greater than 2.5 for the unlabeled PCDD/PCDF ions, and greater than 10.0 for the labeled internal and recovery standards.
- 10.6.6. Response Factor Criteria. The measured RRFs of each analyte and internal standard in the CC3 solution must be within $\pm 30\%$ of the mean RRFs established during initial calibration for method 8280A, and within $\pm 20\%$ of the mean RRFs established during initial calibration for method 8280B,

$$\% \text{Difference} = \frac{(\text{RRF}_i - \text{RRF}_c)}{\text{RRF}_i} \times 100$$

Where:

RRF_i = Relative response factor established during initial calibration.

RRF_c = Relative response factor established during continuing calibration.

- 10.6.7. If any of the criteria listed are not met, the analyst must take corrective actions and reanalyze the continuing calibration standard (CC3). If the criteria are met after corrective action has been taken, then the sample analysis may proceed.
 - 10.6.8. If the criteria are not met after corrective actions have been taken, then a new initial calibration must be performed, beginning with the analysis of the window defining mix if retention times may have been affected. The new initial calibration must meet all of the QC criteria in Section 10.4 before sample analysis may proceed.
- 10.7. Instrument Sensitivity Check
- 10.7.1. In order to demonstrate that the GC/MS/DS system has retained adequate sensitivity during the course of sample analyses, the lowest of the standards (CC1) should be analyzed immediately following the end of each 12-hour sequence during which samples and standards are analyzed. The 12 hour time

limit starts at the time of the injection of the daily calibration solution CC3.
Note: This should be done to show instrument sensitivity criteria has been met during the entire acquisition of the run. If the analysis of the CC1 is delayed (for example a bad injection of the CC1) the CC1 should be analyzed, and the data evaluated for adverse impact. An NCM shall be created to document this anomaly.

- 10.7.2. Analyze a 2 μ L aliquot of the CC1 solution, using the identical instrumental conditions used for analysis of samples and standards.
- 10.7.3. The CC1 solution analyzed immediately following the end of the 12-hour period must meet the following criteria:
 - 10.7.3.1. Retention Time Criteria: The absolute retention time of the recovery standards ^{13}C -1,2,3,4-TCDD and ^{13}C -1,2,3,7,8,9-HxCDD shall not change more than 10 seconds between the initial CC3 analysis and then ending CC1 analysis.
 - 10.7.3.2. All the analytes in the CC1 solution must meet the ion abundance ratio criteria in Table 2.
 - 10.7.3.3. Instrument Sensitivity Criteria: For the CC1 solution, the S/N ratio must be greater than 2.5 for the unlabeled PCDD/PCDF ions and greater than 10.0 for the labeled internal and recovery standards.
- 10.7.4. If the analysis of the CC1 solution at the end of the 12-hour period fails either the ion abundance or S/N criteria above, the laboratory must take corrective action which may include:
 - 10.7.4.1. Performing a new initial calibration, beginning with the analysis if the window defining mix if necessary to set the windows.
 - 10.7.4.2. Start a new analytical sequence
 - 10.7.4.3. Reanalyzing all samples originally analyzed in the preceding 12-hour time period in which:
 - 10.7.4.3.1. No PCDDs/PCDFs were detected, or
 - 10.7.4.3.2. Neither 2,3,7,8-TCDD or 2,3,7,8-TCDF were detected, even if other PCDDs or PCDFs were detected.
 - 10.7.4.4. These reanalyses are necessary because poor S/N ratios indicate a loss of sensitivity that could lead to false negative results, underestimation of concentration, or could cause ion abundance ratios to fall outside the QC limits.

Note: If the samples are not impacted by isotopic ratios outside of

criteria the data can be considered not to be adversely affected and a NCM should be created for this anomaly.

- 10.7.4.5. If the CC1 analysis fails either the ion abundance or S/N criteria, then any samples analyzed during that 12-hour period that indicated the presence of any PCDDs/PCDFs below the method quantitation limit or where EMPC concentrations were reported must be reanalyzed. Samples with positive results above the method quantitation limit need not be reanalyzed.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. GC/MS Analysis

- 11.2.1. Inject a 2 μ L aliquot of the extract into the GC/MS instrument with the following conditions.

Operating Conditions Guidelines	
Column Coating	DB-5 (or SP-2331)
Film Thickness	0.25 μ m
Column Dimensions	60 m x 0.25 mm
Helium Linear Velocity	1.0 mL/min at 28psi
Initial Temperature	190°C
Initial Time	1 min.
Temperature Program	increase to 240°C at 4°C/min. then to 320°C at 20°C /min
Hold Time	until OCDF elutes

- 11.2.2. Analyze the extract by GC/MS and monitor all of the ions listed in Table 2. The same MS parameters used to analyze the calibration solutions shall be used for the sample extracts.

11.3. Dilutions

- 11.3.1. If the concentration in the final extract of any analyte exceeds the upper method calibration limit (MCL) for that compound, the linear range of response versus concentration may have been exceeded. In such cases, the following corrective actions may be undertaken:
 - 11.3.1.1. If the signal for the analyte has saturated the detector, a single dilution and reanalysis of the extract will be made in an attempt to bring the signal within the range of the detector. The reported concentration for the analyte will be qualified appropriately.
 - 11.3.1.2. If the signal for the analyte is above the MCL, but does not saturate the detector, the concentration will be reported and qualified as "E."
 - 11.3.1.3. With the approval of the client, samples may be reextracted and/or reanalyzed with the following adjustments in order to provide a concentration which meets client-specific data quality objectives.
 - 11.3.1.4. Extract and analyze one tenth of the original aliquot. This option is appropriate only if it will provide analyte concentration within the MCL and if the sample aliquot will be representative.
 - 11.3.1.5. Extract an aliquot large enough to be representative, and increase the concentration of internal standard and surrogate spike components added prior to the extraction. The extract is then diluted either prior to or after the clean up procedures.
 - 11.3.1.6. Dilute the original extract. Internal standard components are respiked at an appropriate level prior to analysis. In this case, the internal standard recoveries are taken from the original analysis if possible.
- 11.3.2. An appropriate dilution will result in the largest peak in the diluted sample falling between the mid-point and high-point of the calibration range.
- 11.3.3. Dilutions are performed using an aliquot of the original extract of which approximately 50 μ L remains. Remove an appropriately sized aliquot from the vial and add it to a sufficient volume of tetradecane in a clean 0.3 mL conical vial. For example: Take a 5 μ L aliquot and add 45 μ L of tetradecane for a 10X dilution (dilution factor of 10).
- 11.3.4. The dilution factor is defined as the total volume of sample aliquot and clean solvent divided by the volume of the sample aliquot that was diluted.
- 11.3.5. Inject 2 μ L of the diluted sample extract into the GC/MS and analyze

according to Section 11.2.

- 11.3.6. Diluted samples in which the MS response of any internal standard is <10:1 signal to noise may be quantified using the recovery standard method.

11.3.6.1. The RRF_{rs} method of quantitation is only used when the sample is diluted to the extent that the S/N ratio for the internal standard is less than 10.0 and re-extraction of the sample at a smaller aliquot is not possible.

- 11.4. Polychlorinated Diphenyl Ether (PCDPE) interferences: The identification of a GC peak as a PCDF cannot be made if a signal having S/N greater than 2.5 is detected at the same retention time (± 2 seconds) in the corresponding PCDPE channel (Table 2c). If PCDPE peaks are present, any non 2,3,7,8-isomer peaks with matching retention time (RT) will be eliminated from the calculation of totals for that homologue group. If the PCDPE peak matches the RT of a 2,3,7,8-isomer, the result will be reported as an estimated maximum possible concentration (EMPC).

12. CALCULATIONS/DATA REDUCTION

- 12.1. For a gas chromatographic peak to be unambiguously identified as a PCDD or PCDF, it must meet all of the following criteria:

12.2. Retention Times

- 12.2.1. Retention times are required for all chromatograms; scan numbers are optional. Retention times shall either be printed at the apex of each peak on the chromatogram, or each peak shall be unambiguously labeled with an identifier that refers to the quantitation report, or combination of both shall contain the retention time of each peak and its area.
- 12.2.2. In order to make a positive identification of the 2,3,7,8-substituted isomers for which an isotopically labeled internal or recovery standard is present in the sample extract, the absolute retention time (RT) at the maximum peak height of the analyte must be within -1 to +3 seconds of the retention time of the corresponding labeled standard.
- 12.2.3. In order to make a positive identification of the 2,3,7,8-substituted isomers for which a labeled standard is not available, the relative retention time (RRT) of the analyte must be within 0.05 RRT units of the RRT established by the continuing calibration. The RRT is calculated as follows:

$$RRT = \frac{\text{Retention Time of Analyte}}{\text{Retention Time of Corresponding Internal Standard}}$$

- 12.2.3.1. For non-2,3,7,8-substituted compounds (tetra through hepta), the retention time must be within the retention time windows established by the window defining mix for the corresponding homologue (See Section 10.2).
- 12.3. Peak Identification: All of the specified ions listed in Table 2 for each PCDD/PCDF homologue and labeled standards must be present in the SICP. The ion current response for the two quantitation ions and the M- [COCl]⁺ ions for the analytes must maximize simultaneously (± 2 seconds). This requirement also applies to the internal standards and recovery standards. For the cleanup standard, only one ion is monitored.
- 12.4. Signal-To-Noise Ratio: The integrated ion current for each analyte ion listed in Table 2 must be at least 2.5 times background noise and must not have saturated the detector. The internal standard ions must be at least 10.0 times background noise and must not have saturated the detector. However, if the M-[COCl]⁺ ion does not meet the 2.5 times S/N requirement but meets all other criteria listed in Section 12.1 and, in the judgment of the GC/MS Interpretation Specialist, the peak is a PCDD/PCDF, the peak may be reported as a qualified positive.
- 12.5. Ion Abundance Ratios
- 12.5.1. The relative ion abundance criteria listed in Table 2 for native analytes and internal standards must be met using peak areas to calculate ratios.
- 12.5.2. If interferences are present and ion abundance ratios are not met using peak areas, but all other qualitative identification criteria are met (RT, S/N, presence of all three ions), the chemist may use peak heights to evaluate the ion ratio.
- 12.5.3. If, in the judgment of the analyst, the peak is a PCDD/PCDF, then report the ion abundance ratios determined using peak heights. Quantitate the peaks using peak heights rather than areas for both the target analyte and the internal standard.
- 12.5.4. The identification of a GC peak as a PCDF cannot be made if a signal having S/N greater than 2.5 is detected at the same retention time (± 2 seconds) in the corresponding PCDPE channel (See Table 2). If a PCDPE is detected, an EMPC should be calculated for this GC peak regardless of the ion abundance ratio.
- 12.6. For GC peaks that have met all the identification criteria outlined above, calculate the concentration of the individual PCDD or PCDF isomers using the following formula:

12.6.1. All matrices other than water:

$$\text{Conc. (ug/kg)} = \frac{Q_{is} \times (A_{n1} + A_{n2})}{W \times (A_{is1} + A_{is2}) \times \text{RRF}_n}$$

12.6.2. Water matrices:

$$\text{Conc. (ng/L)} = \frac{Q_{is} \times (A_{n1} + A_{n2})}{V \times (A_{is1} + A_{is2}) \times \text{RRF}_n}$$

Where:

A_{n1}, A_{n2} = integrated ion abundances (peak area) of the quantitation ions of the isomer of interest (Table 2).

A_{is1}, A_{is2} = integrated ion abundances (peak areas) of the quantitation ions of the appropriate internal standard (Table 2).

W = weight of sample extracted, in grams.

V = volume of sample extracted, in liters. Samples are weighed on a balance to two significant figures. Volume is based on the density of water being 1.00 g/mL

Q_{is} = quantity of the appropriate internal standard added to the sample prior to extraction, ng.

RRF_n = calculated relative response factor from continuing calibration (See Section 10.6).

NOTE: In instances where peak heights are used to evaluate ion abundance ratios due to interference (see Section 12.5.3), substitute peak heights for areas in the formula above.

12.6.3. For solid matrices, the units of ng/g that result from the formula above are equivalent to $\mu\text{g/Kg}$. Using isotope dilution techniques for quantitation, the concentration data are recovery corrected, and therefore, the volume of the final extract and the injection volume are implicit in the value of Q_{is} .

12.6.3.1. For homologous series that contain only one 2,3,7,8-substituted isomer (TCDD, PeCDD, HpCDD, OCDD, TCDF, and OCDF), the RRF of the 2,3,7,8-substituted isomer from the continuing calibration (See Section 10.6) will be used to quantitate both the 2,3,7,8-substituted isomers and the non-2,3,7,8-substituted isomers.

12.6.3.2. For homologous series that contain more than one 2,3,7,8-substituted isomer (HxCDD, PeCDF, HxCDF, and HpCDF), the RRF used to calculate the concentration of each 2,3,7,8-substituted isomers will be the RRF determined for that isomer during the continuing calibration (See Section 10.6).

12.6.3.3. For homologous series that contain one or more non-2,3,7,8-substituted isomers, the RRF used to calculate the concentration of these isomers will be the lowest of the RRFs determined during the continuing calibration (See Section 10.6) for the 2,3,7,8-substituted isomers in that homologue. This RRF will yield the highest possible concentration for the non-2,3,7,8-substituted isomers.

NOTE: The relative response factors of given isomers within any homologous series may be different. However, for the purpose of these calculations, it will be assumed that every non-2,3,7,8-substituted isomer for a given homologue level will have the same relative response factor. In order to minimize the effect of this assumption on risk assessment, the 2,3,7,8-substituted isomer with the lowest RRF was chosen as representative of each homologous series. All relative response factor calculations for the non-2,3,7,8-substituted isomers in a given homologue are based on that isomer.

12.7. In addition to the concentrations of specific isomers, the total homologue concentrations are also reported. Calculate the total concentration of each homologous series of PCDDs/PCDFs as follows:

12.7.1. Total Concentration = sum of the concentrations of every positively identified isomer of each PCDD/PCDF homologous series.

12.7.2. The total must include the non-2,3,7,8-substituted isomers as well as the 2,3,7,8-substituted isomers that are also reported separately.

12.8. If the area of any internal standard in a diluted sample is less than 10:1 signal to noise, and re-extraction or post-dilution of internal standards will not improve results the unlabeled PCDD/PCDF concentrations in the sample may be estimated using the formula below. The purpose is to ensure that there is an adequate MS response for quantitation in a diluted sample.

12.8.1. All matrices other than water:

$$\text{Conc. (ug/kg)} = \frac{Q_{rs} \times (A_{n1} + A_{n2}) \times D}{W \times (A_{rs1} + A_{rs2}) \times \text{RRF}_{rs}}$$

12.8.2. Water matrices:

$$\text{Conc. (ng/L)} = \frac{Q_{rs} \times (A_{n1} + A_{n2}) \times D}{V \times (A_{rs1} + A_{rs2}) \times \text{RRF}_{rs}}$$

Where:

D = Dilution factor.

(See Section 10.5 and 12.6 for identification of the rest of the terms.)

12.8.2.1. The RRF_{rs} method of quantitation is only used when the sample is diluted to the extent that the S/N ratio for the internal standard is less than 10.0.

12.9. Report results for soil/sediment, fly ash, and chemical waste samples in micrograms per kilograms ($\mu\text{g/kg}$) or nanograms per gram (ng/g) and water samples in nanograms per liter (ng/L).

12.10. Calculate the percent recovery for each internal standard and the cleanup standard in the sample extract, R_{is} , using the following formula:

$$R_{is} = \frac{Q_{rs} \times (A_{is1} + A_{is2})}{(A_{rs1} + A_{rs2}) \times RRF_{is} \times Q_{is}} \times 100\%$$

(See Sections 10.5.5 and 12.2 for identification of the terms.)

NOTE: When calculating the recovery of the ^{37}Cl -2,3,7,8-TCDD cleanup standard, only one m/z is monitored for this standard; therefore only one peak will be used in the numerator of this formula. Use both peak areas for the ^{13}C -1,2,3,4-TCDD recovery standard in the denominator.

12.11. Sample Specific Reporting Limits (Contract Required Quantitation Limits)

12.11.1. See Table 1 for RL for each PCDF and PCDD analyte based on the nominal sample size amount. The calculation for a true sample size is as follows:

12.11.2. All matrices except water:

$$RL(\text{ug/kg}) = \frac{CC1_{NS} \times FV}{W}$$

12.11.3. Water matrices:

$$RL(\text{ng/L}) = \frac{CC1_{NS} \times FV}{V}$$

Where:

- $CC1_{NS}$ = Concentration of PCDF or PCDD analyte of interest CC1 of the calibration curve.
- FV = The final volume of the extract.
- W = The weight of the solid matrix.
- V = The volume of the aqueous matrix.

12.12. Sample Specific Estimated Detection Limits

12.12.1. The sample specific Estimated Detection Limit (EDL) is the estimate made by the laboratory of the concentration of a given analyte required to produce a

signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, etc.

12.12.2. An EDL is calculated for each 2,3,7,8-substituted isomer that is not identified, regardless of whether or not non-2,3,7,8-substituted isomers in that homologous series are present. The EDL is also calculated for 2,3,7,8-substituted isomers giving responses for both the quantitation ions that are less than 2.5 times the background level.

12.12.3. Using the formula below to calculate an EDL for each absent 2,3,7,8-substituted PCDD/PCDF. The background level (H_x) is determined by measuring the height of the noise at the expected retention times of both quantitation ions of the particular 2,3,7,8-substituted isomer. The expected retention time is determined from the most recent analysis of the CC3 standard on the same GC/MS system.

12.12.3.1. All matrices other than water:

$$\text{EDL (ug/kg)} = \frac{2.5 \times Q \times (H_{x1} + H_{x2})}{W \times (H_{is1} + H_{is2}) \times \text{RRF}_n}$$

12.12.3.2. Water matrices:

$$\text{EDL (ng/L)} = \frac{2.5 \times Q \times (H_{x1} + H_{x2})}{V \times (H_{is1} + H_{is2}) \times \text{RRF}_n}$$

Where:

H_{x1}, H_{x2} = Peak heights of the noise for both of the quantitation ions of the 2,3,7,8-substituted isomer of interest.

H_{is1}, H_{is2} = Peak heights of both the quantitation ions of the appropriate internal standards.

NOTE: If the congener class is non-detect, the largest relative response factor (RRF) for that class will be used to calculate the congener's estimated detection limit.

12.13. Toxicity Equivalency Factor (TEF) Calculation

12.13.1. The 2,3,7,8-TCDD toxicity equivalence of PCDDs/PCDFs present in the sample is calculated according to the method recommended by the Chlorinated Dioxins Workgroup (CDWG) of the EPA and the Centers for Disease Control (CDC). This method assigns a 2,3,7,8-TCDD toxicity equivalency factor to each of the seventeen 2,3,7,8-substituted PCDDs/PCDFs shown in Table 1. The 2,3,7,8-TCDD toxicity equivalence of the PCDDs/PCDFs present in the sample are calculated by summing the product of the TEF and the concentration for each of the compounds listed in Table 1.

- 12.13.2. The exclusion of homologues such as mono-, di-, tri-, and the non-2,3,7,8-substituted isomers in the higher homologous series do not mean that they are not toxic. Their toxicity, as estimated at this time, is much less than the toxicity of the compounds listed in Table 1. Hence, only 2,3,7,8-substituted isomers are included in the TEF calculations. The procedure for calculating the 2,3,7,8-TCDD toxic equivalence cited above is not claimed by the CDWG to be based on a thoroughly established scientific foundation. Rather, the procedure represents a "Consensus Recommendation on Science Policy."
- 12.13.3. When calculating the 2,3,7,8-TCDD toxicity equivalence of a sample, include only those 2,3,7,8-substituted isomers that were detected in the sample and met all of the qualitative identification criteria in Section 11.8. Do not include EMPC or EDL values in the TEF calculations.
- 12.14. Due to a variety of situations that may occur during method performance, the laboratory is required to reextract and reanalyze certain samples or groups of samples. Except in the case of dilutions, the term "rerun" shall indicate sample extraction, cleanup, and reanalysis. When dilutions are required, the original extract shall be diluted and reanalyzed.
- 12.14.1. When the rerun is required due to matrix effects, interferences, or other problems encountered, the client, upon authorization to perform, will pay the laboratory for the reruns. When the rerun is required due to laboratory materials, equipment or instrumentation problems, or lack of laboratory adherence to specified method procedures, then the rerun shall not be billable.
- 12.14.2. The following sample reruns may be required as defined below:
- 12.14.2.1. If the original sample has a percent recovery of any internal standard or the cleanup standard outside of the range of 25-150%, then re-extraction and reanalysis may be required. If signal to noise of the internal standards is >10:1 the samples should be evaluated for possible adverse impact. If deemed unacceptable the samples shall be re-extracted upon client approval.
- 12.14.2.2. If the internal standards are not present with at least a 10:1 S/N ratio at their respective m/z's (316, 318, 332, 334, 402, 404, 420, 422, 470, and 472), then re-extraction and reanalysis are required. If the ³⁷Cl-2,3,7,8-TCDD is not present with at least a 10/1 S/N ratio at m/z 328, then re-extraction and reanalysis may be required.
- 12.14.3. If the calculated concentration of the unlabeled PCDDs/PCDFs exceeded the initial calibration range, the sample may be diluted and reanalyzed.

Note: Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

13. METHOD PERFORMANCE

- 13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.
- 13.2. Method Detection Limit
The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.
- 13.3. Initial Demonstration
The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.
 - 13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.
 - 13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.
- 13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- 13.5. It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. Section 10 provides recommended GC conditions that can be used to satisfy the required criteria.
- 13.6. The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

- 15.1. Autovials containing tetradecane extracts. As the autovials are removed from the instrument after analysis, they are collected in archive boxes and retained pending additional instructions. When no longer needed, the archive boxes are emptied into vial drums. When full or after no more than one year, the vial drums are moved to the waste disposal area for disposal as PCB waste.

16. REFERENCES/CROSS REFERENCES

- 16.1. Document Number DFLM 01.0, including Revision DFLM 01.1 (September 1991).
- 16.2. Update of Toxicity of Equivalency Factors (TEFs) for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-dioxins and Dibenzofurans (CDDs/CDFs), EPA 625/3-89/016 (March 1989).
- 16.3. SW-846, Method 8280A, Update III, December 1996.
- 16.4. SW-846, Method 8280B, Revision 1, February 2007.
- 16.5. WS-QA-0023, Nonconformance and Corrective Action System

17. METHOD MODIFICATIONS

Unless otherwise specified, the modifications below apply to both method 8280A and method 8280B.

- 17.1. The levels of pentachlorinated dioxins and furans are at 0.25, 0.625, 1.25, 2.5 and 5.0 in the calibration solutions as opposed to the method specified levels of 0.1, 0.25, 0.5, 1.0 and 2.0. (See Table 3) This adjustment aligns the calibration solutions with the RLs.

- 17.2. The levels of the hexa- to heptachlorinated isomers were adjusted from 1.2 ng/μL to 0.5 ng/μL in the window defining mixture. (See Table 4)
- 17.3. The stock solution concentrations and volumes added to extracts were adjusted for ease of application.
- 17.4. RL convention has been added in Section 12.7 to be used as a Lower Calibration Reporting Limit instead of reporting each analyte to zero using the EDL calculation in Section 12.8. This will give an optional reporting technique.
- 17.5. When any internal standard recovery is outside the 25-150% window, the reference methods state that re-extraction and reanalysis are necessary. This SOP uses a 10:1 signal to noise criterion to determine whether a re-extraction is necessary.
- 17.6. Method 8280A only: All analytes are present in each calibration level, not just in the CC3 level. Consequently, RRFs and RSDs are calculated for each unlabelled compound.

18. ATTACHMENTS

- 18.1. Table 1 – Target Compound List and Reporting Limits
- 18.2. Table 2 – Criteria for Isotopic Ratio Measurements for PCDDs/PCDFs
- 18.3. Table 3 – Concentration of Calibration Solutions
- 18.4. Table 4 – PCDD/PCDF Isomers in the Window Defining Mix for a 60M DB-5 (or equivalent) Column
- 18.5. Table 5 – Matrix Spiking Solution
- 18.6. Table 6 – Internal Standard, Recovery Standard, and Cleanup Standard Solutions

19. REVISION HISTORY

- 19.1. WS-ID-0011, Revision 4, Effective 10/03/2008
 - 19.1.1. Updated to address running the method on a HiRes instrument.
 - 19.1.2. Editorial changes.
 - 19.1.3. Changes made to incorporate Method 8280B.
- 19.2. WS-ID-0011, Revision 3.2, Effective 4/09/2008
 - 19.2.1. Added South Carolina rule to prepare an MS/MSD with every batch.

- 19.3. WS-ID-0011, Revision 3.1, Effective 6/27/2007
 - 19.3.1. Updated to TestAmerica format.
- 19.4. SAC-ID-0011, Revision 3.1, Effective 6/27/2007
 - 19.4.1. Updated to comply with DoD QSM.
- 19.5. SAC-ID-0011, Revision 3, Effective 1/31/2007
 - 19.5.1. The preparation portion of the method was removed from this SOP into a separate SOP, SAC-IDP-0011.
- 19.6. SAC-ID-0011, Revision 2, Effective 5/5/2003
- 19.7. SAC-ID-0011, Revision 1.1, Effective 12/7/2001
- 19.8. SAC-ID-0011, Revision 1, Effective 10/15/1998

Table 1
Target Compound List (TCL) and Reporting Limits¹ (RL)

PCDD/PCDF	CAS Number	Water (ng/L) 1L Sample Size	Soil (µg/Kg) 10g Sample Size	Chemical Waste (µg/Kg) 0.1g Sample Size
2,3,7,8-TCDD	1746-01-6	5	0.5	50
2,3,7,8-TCDF	51207-31-9	5	0.5	50
1,2,3,7,8-PeCDF	57117-41-6	12.5	1.25	125
1,2,3,7,8-PeCDD	40321-76-4	12.5	1.25	125
2,3,4,7,8-PeCDF	57117-31-4	12.5	1.25	125
1,2,3,4,7,8-HxCDF	70648-26-9	12.5	1.25	125
1,2,3,6,7,8-HxCDF	57117-44-9	12.5	1.25	125
1,2,3,4,7,8-HxCDD	39227-28-6	12.5	1.25	125
1,2,3,6,7,8-HxCDD	57653-85-7	12.5	1.25	125
1,2,3,7,8,9-HxCDD	19408-74-3	12.5	1.25	125
2,3,4,6,7,8-HxCDF	60851-34-5	12.5	1.25	125
1,2,3,7,8,9-HxCDF	72918-21-9	12.5	1.25	125
1,2,3,4,6,7,8-HpCDF	67562-39-4	12.5	1.25	125
1,2,3,4,6,7,8-HpCDD	35822-46-9	12.5	1.25	125
1,2,3,4,7,8,9-HpCDF	55673-89-7	12.5	1.25	125
OCDD	3268-87-9	25	2.5	250
OCDF	39001-02-0	25	2.5	250

¹ All RL values listed here are based on the wet weight of the sample.

Table 2a: LRMS Criteria for Isotopic Ratio Measurements for PCDDs/PCDFs

Analyte	Associated Internal Standard or recovery standard	Selected Ions	M-[COCl] ⁺	Theoretical Ion Abundance	Control Limits
TCDD	¹³ C-2,3,7,8-TCDD	320/322	259	0.77	0.65-0.89
PeCDD	¹³ C-2,3,7,8-TCDD	356/358	293	1.55	1.32-1.78
HxCDD	¹³ C-1,2,3,6,7,8-HxCDD	390/392	327	1.24	1.05-1.43
HpCDD	¹³ C-1,2,3,6,7,8-HxCDD	424/426	361	1.04	0.88-1.20
OCDD	¹³ C-OCDD	458/460	395	0.89	0.76-1.02
TCDF	¹³ C-2,3,7,8-TCDF	304/306	243	0.77	0.65-0.89
PeCDF	¹³ C-2,3,7,8-TCDF	340/342	277	1.55	1.32-1.78
HxCDF	¹³ C-1,2,3,4,7,8,9-HpCDF	374/376	311	1.24	1.05-1.43
HpCDF	¹³ C-1,2,3,4,7,8,9-HpCDF	408/410	345	1.04	0.88-1.20
OCDF	¹³ C-OCDD	442/444	379	0.89	0.76-1.02
Internal Standards					
¹³ C-2,3,7,8-TCDD	¹³ C-1,2,3,4-TCDD	332/334		0.77	0.65-0.89
¹³ C-1,2,3,7,8-PeCDD ⁽¹⁾		368/370		1.55	1.32-1.78
¹³ C-1,2,3,4,7,8-HxCDD ⁽¹⁾		402/404		1.24	1.05-1.43
¹³ C-1,2,3,6,7,8-HxCDD	¹³ C-1,2,3,7,8,9-HxCDD	402/404		1.24	1.05-1.43
¹³ C-1,2,3,4,6,7,8-HpCDD ⁽¹⁾		436/438		1.04	0.88-1.20
¹³ C-OCDD	¹³ C-1,2,3,7,8,9-HxCDD	470/472		0.89	0.76-1.01
¹³ C-2,3,7,8-TCDF	¹³ C-1,2,3,4-TCDD	316/318		0.77	0.65-0.89
¹³ C-1,2,3,7,8-PeCDF ⁽¹⁾		352/354		1.55	1.32-1.78
¹³ C-2,3,4,7,8-PeCDF ⁽¹⁾		352/354		1.55	1.32-1.78
¹³ C-1,2,3,4,7,8-HxCDF ⁽¹⁾		384/386		0.51	0.43-0.59
¹³ C-1,2,3,6,7,8-HxCDF ⁽¹⁾		384/386		0.51	0.43-0.59
¹³ C-2,3,4,6,7,8-HxCDF ⁽¹⁾		384/386		0.51	0.43-0.59
¹³ C-1,2,3,7,8,9-HxCDF ⁽¹⁾		384/386		0.51	0.43-0.59
¹³ C-1,2,3,4,6,7,8-HpCDF	¹³ C-1,2,3,7,8,9-HxCDD	420/422		1.04	0.88-1.20
Recovery Standards					
¹³ C-1,2,3,4-TCDD		332/334		0.77	0.65-0.89
¹³ C-1,2,3,7,8,9-HxCDD		402/404		1.24	1.05-1.43
Clean up Standards					
³⁷ Cl-2,3,7,8-TCDD ⁽²⁾	¹³ C-1,2,3,4-TCDD	328			

⁽¹⁾ These extra internal standards are not mentioned in Method 8280A. These may be used to improve the method ruggedness and if interferences from these in other ions are not seen or significant.

⁽²⁾ There is only one quantitation ion monitored for the cleanup standard.

Table 2b: HRMS Criteria for Isotopic Ratio Measurements for PCDDs/PCDFs

Analyte	Associated Internal Standard or recovery standard	Selected Ions	M-[COCl] ⁺	Theoretical Ion Abundance	Control Limits
TCDD	¹³ C-2,3,7,8-TCDD	319.8965/321.8936	258.9298	0.77	0.65-0.89
PeCDD	¹³ C-2,3,7,8-TCDD	355.8546/357.8516	290.8938	1.55	1.32-1.78
HxCDD	¹³ C-1,2,3,6,7,8-HxCDD	389.8157/391.8127	326.8519	1.24	1.05-1.43
HpCDD	¹³ C-1,2,3,6,7,8-HxCDD	423.7766/425.7737	360.8129	1.04	0.88-1.20
OCDD	¹³ C-OCDD	457.7377/459.7348	394.7739	0.89	0.76-1.02
TCDF	¹³ C-2,3,7,8-TCDF	303.9016/305.8987	240.9379	0.77	0.65-0.89
PeCDF	¹³ C-2,3,7,8-TCDF	339.8597/341.8567	276.8959	1.55	1.32-1.78
HxCDF	¹³ C-1,2,3,4,7,8,9-HpCDF	373.8208/375.8178	310.8570	1.24	1.05-1.43
HpCDF	¹³ C-1,2,3,4,7,8,9-HpCDF	407.7818/409.7789	344.8180	1.04	0.88-1.20
OCDF	¹³ C-OCDD	441.7428/443.7399	378.7790	0.89	0.76-1.02
Internal Standards					
¹³ C-2,3,7,8-TCDD	¹³ C-1,2,3,4-TCDD	331.9368/333.9339		0.77	0.65-0.89
¹³ C-1,2,3,7,8-PeCDD ⁽¹⁾		367.8949/369.8919		1.55	1.32-1.78
¹³ C-1,2,3,4,7,8-HxCDD ⁽¹⁾		401.8559/403.8529		1.24	1.05-1.43
¹³ C-1,2,3,6,7,8-HxCDD	¹³ C-1,2,3,7,8,9-HxCDD	401.8559/403.8529		1.24	1.05-1.43
¹³ C-1,2,3,4,6,7,8-HpCDD ⁽¹⁾		435.8169/437.8140		1.04	0.88-1.20
¹³ C-OCDD	¹³ C-1,2,3,7,8,9-HxCDD	469.7779/471.7750		0.89	0.76-1.01
¹³ C-2,3,7,8-TCDF	¹³ C-1,2,3,4-TCDD	315.9419/317.9389		0.77	0.65-0.89
¹³ C-1,2,3,7,8-PeCDF ⁽¹⁾		351.9000/353.8970		1.55	1.32-1.78
¹³ C-2,3,4,7,8-PeCDF ⁽¹⁾		351.9000/353.8970		1.55	1.32-1.78
¹³ C-1,2,3,4,7,8-HxCDF ⁽¹⁾		383.8639/385.8610		0.51	0.43-0.59
¹³ C-1,2,3,6,7,8-HxCDF ⁽¹⁾		383.8639/385.8610		0.51	0.43-0.59
¹³ C-2,3,4,6,7,8-HxCDF ⁽¹⁾		383.8639/385.8610		0.51	0.43-0.59
¹³ C-1,2,3,7,8,9-HxCDF ⁽¹⁾		383.8639/385.86106		0.51	0.43-0.59
¹³ C-1,2,3,4,6,7,8-HpCDF	¹³ C-1,2,3,7,8,9-HxCDD	417.8253/419.8220		0.44	0.37-0.51
Recovery Standards					
¹³ C-1,2,3,4-TCDD		331.9368/333.9339		0.77	0.65-0.89
¹³ C-1,2,3,7,8,9-HxCDD		401.8559/403.8529		1.24	1.05-1.43
Clean up Standards					
³⁷ Cl-2,3,7,8-TCDD ⁽²⁾	¹³ C-1,2,3,4-TCDD	327.8847			

⁽¹⁾ These extra internal standards are not mentioned in Method 8280A. These may be used to improve the method ruggedness and if interferences from these in other ions are not seen or significant.

⁽²⁾ There is only one quantitation ion monitored for the cleanup standard.

Table 2c: Masses for Diphenyl Ether Interference Identification

Analyte	Selected Ion HRMS	Selected Ion LRMS
TCDF	375.8364	376
PeCDF	409.7974	410
HxCDF	445.7555	446
HpCDF	479.7165	480
OCDF	513.6775	514

Table 3
Concentration of Calibration Solutions (ng/μL)

Analyte	CC1	CC2	CC3	CC4	CC5
2,3,7,8-TCDD	0.1	0.25	0.5	1.0	2.0
2,3,7,8-TCDF	0.1	0.25	0.5	1.0	2.0
1,2,3,7,8-PeCDF	0.25	0.625	1.25	2.0	5.0
1,2,3,7,8-PeCDD	0.25	0.625	1.25	2.0	5.0
2,3,4,7,8-PeCDF	0.25	0.625	1.25	2.0	5.0
1,2,3,4,7,8-HxCDF	0.25	0.625	1.25	2.0	5.0
1,2,3,6,7,8-HxCDF	0.25	0.625	1.25	2.5	5.0
1,2,3,4,7,8-HxCDD	0.25	0.625	1.25	2.5	5.0
1,2,3,6,7,8-HxCDD	0.25	0.625	1.25	2.5	5.0
13C-1,2,3,4-TCDD	0.5	0.5	0.5	0.5	0.5
13C-1,2,3,7,8,9-HxCDD	0.5	0.5	0.5	0.5	0.5
1,2,3,4,7,8,9-HpCDF	0.25	0.625	1.25	2.5	5.0
1,2,3,4,6,7,8-HpCDF	0.25	0.625	1.25	2.5	5.0
1,2,3,4,6,7,8-HpCDD	0.25	0.625	1.25	2.5	5.0
OCDD	0.5	1.25	2.5	5.0	10.0
OCDF	0.5	1.25	2.5	5.0	10.0
¹³ C-2,3,7,8-TCDD	0.5	0.5	0.5	0.5	0.5
¹³ C-2,3,7,8-TCDF	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,7,8-PeCDF ⁽¹⁾	0.5	0.5	0.5	0.5	0.5
¹³ C-2,3,4,7,8-PeCDF ⁽¹⁾	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,7,8-PeCDD ⁽¹⁾	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,4,7,8-HxCDF ⁽¹⁾	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,6,7,8-HxCDF ⁽¹⁾	0.5	0.5	0.5	0.5	0.5
¹³ C-2,3,4,6,7,8-HxCDF ⁽¹⁾	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,7,8,9-HxCDF ⁽¹⁾	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,4,7,8-HxCDD ⁽¹⁾	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,6,7,8-HxCDD	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,4,6,7,8-HpCDF	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,4,6,7,8-HpCDD ⁽¹⁾	0.5	0.5	0.5	0.5	0.5
¹³ C-OCDD	1.0	1.0	1.0	1.0	1.0
¹³ C-1,2,3,4-TCDD	0.5	0.5	0.5	0.5	0.5
¹³ C-1,2,3,7,8,9-HxCDD	0.5	0.5	0.5	0.5	0.5
³⁷ Cl-2,3,7,8-TCDD	0.25	0.25	0.25	0.25	0.25

⁽¹⁾ These extra internal standards are not mentioned in Method 8280A. These may be used to improve the method ruggedness and if interferences from these in other ions are not seen or significant.

Table 4
PCDD/PCDF Isomers in the Window Defining Mix for A 60M DB-5 (or equivalent) Column

Homologue	First Eluted	Last Eluted	Approximate Concentration (ng/ μ L)
TCDD	1,3,6,8-	1,2,8,9-	0.5
TCDF	1,3,6,8-	1,2,8,9-	0.5
PeCDD	1,2,4,7,9-	1,2,3,8,9-	0.5
PeCDF	1,3,4,6,8-	1,2,3,8,9-	0.5
HxCDD	1,2,4,6,7,9-	1,2,3,4,6,7-	0.5
HxCDF	1,2,3,4,6,8-	1,2,3,4,8,9-	0.5
HpCDD	1,2,3,4,6,7,9-	1,2,3,4,6,7,8-	0.5
HpCDF	1,2,3,4,6,7,8-	1,2,3,4,7,8,9-	0.5


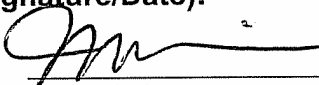
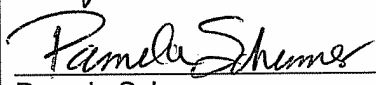
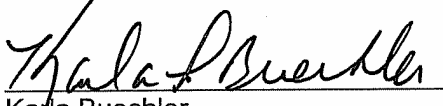
Table 5
Matrix Spiking Solution

Analyte	Concentration (ng/ μ L)
2,3,7,8-TCDD	0.5
2,3,7,8-TCDF	0.5
1,2,3,7,8-PeCDF	1.25
1,2,3,7,8-PeCDD	1.25
1,2,3,6,7,8-HxCDF	1.25
1,2,3,6,7,8-HxCDD	1.25
1,2,3,4,6,7,8-HpCDF	1.25
1,2,3,4,6,7,8-HpCDD	1.25
OCDD	2.5
OCDF	2.5

This solution is prepared in isooctane (or nonane) and diluted with acetone prior to use. Only the ten isomers listed above will be used for evaluation. All seventeen 2,3,7,8-substituted isomers are incorporated into the matrix spike solution.

**Title: EXTRACTION OF POLYCHLORINATED DIBENZO-P-DIOXINS
AND DIBENZOFURANS FOR LOW-RESOLUTION GC/MS ANALYSIS**

[Methods 8280A & 8280B]

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1. SCOPE AND APPLICATION

- 1.1. This procedure is appropriate for the extraction of tetra-, penta-, hexa-, hepta-, and octachlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) in water, soil, sediment, fly ash, and chemical waste samples in accordance with methods 8280A and 8280B. Sample extracts are subjected to specified cleanup procedures.
- 1.2. The analytical method requires the use of high resolution gas chromatography and low resolution mass spectrometry per methods 8280A or 8280B, as detailed in SOP WS-ID-0011.

2. SUMMARY OF METHOD

- 2.1. A specified amount of water, soil, sediment, fly ash or chemical waste samples is spiked with the internal standard solution. Soil, sediment, fly ash or chemical waste samples are extracted with toluene in a combination of Soxhlet extractor (or equivalent Soxtherm system) with a Dean Stark water separator (SDS). Waste samples that fully dissolve in an organic solvent are diluted, then proceed to clean up. Aqueous samples are filtered prior to extraction, and the glass fiber (or cellulose) filter paper extracted as a solid. The filtered aqueous fraction is extracted with methylene chloride using a separatory funnel.
- 2.2. Following a solvent exchange step, the extract may be cleaned up by a variety of column chromatographic procedures (including acid-base washing treatment, silica gel, acid alumina, and carbon/silica columns) to eliminate sample components that may interfere with the detection and measurement of PCDDs/PCDFs. Immediately prior to cleanup, all extracts are spiked with a cleanup standard. The recovery standards are added to the aliquot and the extract is reduced to a final volume of 50 μ L of tetradecane.

3. DEFINITIONS

- 3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).
- 3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.
- 3.3. Internal Standards: Internal standards are isotopically labeled analogs which are present at the same concentration in every blank, field sample, quality control sample, and calibration solution. The internal standards are added to the sample before extraction and are used to measure the concentrations of the analytes.

4. INTERFERENCES

- 4.1. Sample components may interfere with the detection and measurement of PCDDs/PCDFs. To reduce interferences, the extract may be cleaned up by a variety of column chromatographic procedures.
- 4.2. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines which may cause misinterpretation of the chromatographic data. All of these materials shall be demonstrated to be free from interferences under the conditions of analysis by running laboratory method blanks. Analysts must avoid using PVC gloves due to the possibility of contamination.
- 4.3. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all glass systems may be necessary.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

- 5.1.1. When Dean-Stark/Soxhlet clean-ups or extractions are performed overnight or unattended, special precautions must be taken. Open the chiller valves to the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.
- 5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

- 5.1.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.1.4. Laboratory procedures such as the repetitive use of pipettes, the repetitive transferring of extracts and the manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.
- 5.1.5. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed.
- 5.1.6. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar or similar cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.
- 5.1.7. Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.
- 5.1.8. The use of vacuum systems during rotovap concentration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.
- 5.2. Primary Materials Used
The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Cyclohexane	Flammable Irritant	300 ppm TWA	Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric acid (1)	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Isooctane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
Sodium hydroxide	Corrosive Poison	2 ppm, 5 mg/m ³	This material will cause burns if it comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric acid (1)	Corrosive Oxidizer Dehydrator	1 mg/m ³	This material will cause burns if it comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Filters

- 6.1.1. Glass wool, methylene chloride rinsed
- 6.1.2. Glass fiber filter (or equivalent): 15 cm
- 6.1.3. Whatman #1 (or equivalent)
- 6.1.4. White quartz sand. 60/70 mesh, used in the SDS extractor.

6.2. Glass wool, silanized.

6.3. Miscellaneous clean glassware

6.4. Dean Stark water separator apparatus.

6.5. Concentrator tubes, 19 mm x 150 mm culture tubes (or equivalent).

6.6. Separatory funnels, 500 mL and 2 L separatory funnels with a Teflon stopcock.

6.7. Soxhlet apparatus, 500 mL flask, all glass - Complete with glass extractor body, condenser, heating mantle and variable transformer for heat control.

6.8. Teflon® boiling chips

6.9. Buchner funnel, 15 cm

6.10. Filtration flask, 1 L capacity (if needed)

7. REAGENTS AND STANDARDS

- 7.1. Solvents: High purity, distilled-in-glass: hexane, methanol, methylene chloride, toluene, isooctane, cyclohexane, acetone, and tetradecane.

- 7.2. Sodium sulfate: granular, anhydrous: rinse with methylene chloride.
- 7.3. Sodium hydroxide, ACS grade
 - 7.3.1. Prepare a 10 N solution in distilled water.
 - 7.3.2. Prepare a 1 N solution in distilled water.
- 7.4. Sulfuric acid, concentrated ACS grade, specific gravity 1.84
- 7.5. Hydrochloric acid, concentrated ACS grade, specific gravity 1.17. Prepare a 1 N solution in distilled water for pretreatment of fly ash samples.
- 7.6. Column chromatography reagents
 - 7.6.1. Alumina, acidic: ICN activated AA, Super I, ICN Biomedical or equivalent
 - 7.6.2. Activated carbon: EM Science or equivalent. Carbon is cleaned by rinsing with methanol and drying in an oven. Store dry, cleaned carbon in Teflon lined clear glass jar at room temperature.
 - 7.6.3. Silica gel: high purity grade silica gel 60, 70-230 mesh or equivalent. Activate for up to 12 hours at 190°C before use. Store at 190°C in a covered flask.
 - 7.6.4. Carbon/silica gel: Mix 5.0 g carbon and 95 g activated silica gel to yield a 5% w/w carbon on silica (other weights may be used to achieve 5% w/w carbon on silica). Store in a covered jar in a cool dry location.
 - 7.6.5. H₂SO₄ silica gel: Mix 44 g concentrated H₂SO₄ and 56 g activated silica gel to yield a 44% w/w H₂SO₄ on silica (other weights may be used to achieve 44% w/w H₂SO₄ on silica). Stir and shake until free flowing. Store at room temperature.
- 7.7. Internal standard solution: The solution contains at least five internal standards in isooctane at the nominal concentrations listed in Table 2. Extra Internal Standards may include some or all of the additional labeled isomers listed in Table 2 to increase method ruggedness. Stock solutions have a 10 year expiration date. Working standards have a 1 year expiration date, and may be re-verified.
- 7.8. Recovery standard solution: The isooctane solution contains the recovery standards ¹³C-1,2,3,4-TCDD and ¹³C-1,2,3,7,8,9-HxCDD at concentrations of 0.5 ng/μL. Recovery standards are used to measure the recovery of the internal standards. Stock solutions have a 10 year expiration date. Working standards have a 1 year expiration date, and may be re-verified.

- 7.9. The cleanup standard is a solution containing ^{37}Cl -2,3,7,8-TCDD at a concentration of 0.25 ng/ μL (0.25 $\mu\text{g/mL}$) in isooctane and is added to all sample extracts prior to cleanup. The recovery of this compound is used to judge the efficiency of the cleanup procedures. Stock solutions have a 10 year expiration date. Working standards have a 1 year expiration date, and may be re-verified.
- 7.10. The matrix spiking standard is a solution containing 10 of the 2,3,7,8-substituted isomers at the concentrations listed in Table 1 in isooctane, and is used to prepare the spiked sample aliquots. Dilute 50 μL of this standard to 1.0 mL with acetone and add to the aliquot chosen for spiking. Stock solutions have a 10 year expiration date. Working standards have a 1 year expiration date, and may be re-verified.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Protect samples from light at the time of receipt until extraction in order to minimize the potential for photodecomposition.
- 8.2. All samples should be stored at $4^\circ \pm 2^\circ\text{C}$ in the dark and extracted within 30 days of collection and completely analyzed within 45 days of extraction. If samples are analyzed after the holding time expiration date, the results should be flagged appropriately.
- 8.3. For method 8280B, samples should be stored at $\leq 6^\circ\text{C}$ in the dark, and extracted within 30 days of collection, with analysis within 45 days of extraction.
- 8.4. For method 8280B, sample extracts should be stored at $\leq 6^\circ\text{C}$ in the dark.

NOTE: The holding times listed in above are recommendations, as noted by the method. PCDDs and PCDFs are very stable in a variety of matrices, and holding times under the conditions listed above may be as high as a year for certain matrices. Sample extracts, however, should always be analyzed within 45 days of extraction.

9. QUALITY CONTROL

- 9.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented in a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when internal standard recoveries are outside control limits. Re-extraction of the blank, other batch QC and the affected samples are required when the method blank is deemed unacceptable. Refer to Policy WS-PQA-0003 for specific acceptance criteria.

Certain programs, such as DOD QSM, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than $\frac{1}{2}$ the lower calibration limit or the reporting limit.

- 9.1.1. Re-extraction of the blank, QC and affected samples for some matrices including wipes are generally not possible because the entire sample is consumed in the initial extraction. Re-processing of the archive is the only available check of the process in this case.
- 9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented in a Non-Conformance memo, then implemented when the recoveries of any spiked analyte is outside control limits. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. Refer to policy WS-PQA-0003 for specific acceptance criteria.
- 9.3. The assessment of matrix effects on method performance, as required by NELAP, is met, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance can be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted at the client's request only. An exception to this rule is a batch containing South Carolina samples. These batches must have an MS/MSD prepared. The MS/MSD are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recovery of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.
 - 9.3.1. Matrix Spike (MS): A sample that is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined and used to estimate the effect of the sample matrix upon the analytical methodology.
 - 9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

- 9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").
 - 9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.
 - 9.3.5. Analyze the MS and MSD samples as described in Section 11.
 - 9.3.6. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.
- 9.4. Duplicates
- 9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10 g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.
- 9.5. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

10. CALIBRATION

- 10.1. On a monthly basis, calibrate any auto-pipettors to be used in accordance with SOP WS-QA-0004.
- 10.2. On a daily basis, calibrate any balances to be used in accordance with SOP WS-QA-0041.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Soxhlet/Soxtherm-Dean Stark (SDS) Pre-Extraction

11.2.1. Do not bake the components of the SDS apparatus as part of routine cleaning. Repeated baking of glassware can cause active sites on the glass surface to absorb PCDDs/PCDFs and other analytes. All glass parts of the SDS apparatus, including the thimbles (cellulose only), must be pre-extracted with toluene for approximately three hours prior to use. For the Soxtherm, the cleaning cycle can be anywhere from 1 hour to 3 hours based on QC results from high level samples. Pre-extraction will ensure that the glassware is as clean as possible and minimize cross contamination problems. Discard the used toluene, or pool it for later analysis to verify the cleanliness of the glassware.

11.2.1.1. The extraction of soil sediment, ash, and filtered particulates from water samples will require the use of a Soxhlet thimble (or equivalent).

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensers are cold before you turn the heating element on. Check all of the condensers about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the last chemist to leave the department is to check all condensers to ensure that they are cold and functioning properly, and the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.2.2. After pre-extraction for at least three hours, allow the apparatus to cool and remove the thimble. Prepare the cleaned thimble with approximately 10 g of cleaned quartz sand (for soil/sediment and ash samples only).

11.2.2.1. For South Carolina samples only, the extraction of soil/sediment, fly ash, and particulates from water samples will require the use of a Soxhlet thimble. See Section 11.2.1 for a discussion of pre-extraction of glassware such as the SDS. Prior to pre-extraction, prepare the thimble by adding 5 g of 70/230 mesh silica gel to the thimble to produce a thin layer in the bottom of the thimble. This layer will trap fine particles in the thimble. Add 80 – 100 g of quartz sand on top of the silica gel, and place the thimble in the extractor.

11.2.2.2. For South Carolina samples only, pre-extract the SDS for 3 hr. with toluene, then allow the apparatus to cool and remove the thimble.

Mix the appropriate weight of sample with the sand in the thimble, being careful not to disturb the silica gel layer.

11.2.2.3. If the sample aliquot to be extracted contains large lumps or is otherwise not easily mixed in the thimble, quartz sand can be added to the sample and mixed in another container to break up the large lumps.

11.2.2.4. If a sample with particularly high moisture content is to be extracted, it may be helpful to leave a small conical depression in the material in the thimble. This procedure will allow the water to drain through the thimble more quickly during the early hours of the extraction. As the moisture is removed during the first few hours of extraction, the depression will collapse, and the sample can be uniformly extracted.

11.3. Chemical Waste Sample Extraction

11.3.1. If a waste sample completely dissolves in an organic solvent, then this section may be skipped. Proceed as described in Section 11.3.4.

11.3.2. Assemble a flask (500 mL), a Dean Stark trap, and a condenser, and pre-extract per Section 11.2.

11.3.3. Oily Sludge/Wet Fuel Oil: Weigh about 1 g (or appropriate sample size) of sample to two decimal places into a tared pre-extracted 500 mL flask. Add internal standard solution (Table 2) to the sample in the flask. Attach the pre-extracted Dean Stark water separator and condenser to the flask, and extract the sample by refluxing it with approximately 100 mL of toluene for at least three hours.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensers are cold before proceeding. Check all of the condensers about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cycle is to be left unattended (e.g., overnight) the last chemist to leave the department is to check all condensers to ensure that they are cold and functioning properly, and the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.3.3.1. Continue refluxing the sample until all the water has been removed. Cool the sample, and filter the toluene extract through a rinsed glass fiber filter into a 500 mL round bottom flask. Rinse the filter with 10 mL of toluene, and combine the extract and rinsate. Concentrate the combined solution to less than 2 mL using a rotary evaporator as described in Section 11.6.2.

- 11.3.4. Waste Dilution: Weigh 1 g sample to 2 decimal places in a VOA vial. Add 40 mL of a suitable solvent to dissolve the waste. Mix. Remove 4 mL of the solution and spike with internal standard. If MS/SD aliquots are requested, spike these aliquots with the appropriate amounts of native solution. Proceed to cleanups in Section 11.7.

Note: Different initial sample sizes and solvent volumes can be used provided the final aliquot taken to cleanups contains 0.1 g of the sample or as required by expected analyte concentration or client request.

11.4. Soil/Sediment/Filter Sample Extraction

- 11.4.1. An extremely wet sample may require centrifugation to remove standing water before extraction.
- 11.4.2. Weigh about 10 g of soil to two decimal places and transfer to a pre-extracted thimble (See Section 11.2). Mix the sample with the quartz sand and add internal standard solution (See Table 2) to the sample/sand mixture. Add small portions of the solution at several sites on the surface of the sample/sand mixture.
- 11.4.2.1. For the filtered particulate portion of aqueous samples (See Section 11.5.1.1), add the filter to the cleaned extraction thimble into a pre-extracted SDS apparatus. Do not add additional internal standard.
- 11.4.3. Place the thimble in the SDS apparatus. Add 200 mL-300 mL toluene to the SDS apparatus and reflux for 16 hours. The solvent must cycle completely through the system 5-10 times per hour.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensers are cold before proceeding. Check all of the condensers about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cycle is to be left unattended (e.g., overnight) the last chemist to leave the department is to check all condensers to ensure that they are cold and functioning properly, and the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

- 11.4.4. Alternative extraction using Soxtherm: Add approximately 175 mL of toluene to the extraction beaker and set the apparatus to boil for 1 hour, reduce solvent to 90 mL, reflux for an additional 1 hour, reduce solvent to 10 mL to 20 mL, then cool for approximately 30 minutes.
- 11.4.5. Alternative extraction using Soxtech: Add approximately 175 mL of toluene to the extraction beaker and set the apparatus to boil for 1 hour, lift thimble from solvent, reflux for an additional 1 hour, then cool for approximately 30 minutes.

11.4.6. Estimate the percent solids content using WS-OP-0013, Determination of Percent Moisture.

11.4.7. Concentrate this extract as described in Section 11.6.

11.4.7.1. For Aqueous filter portion, combine filter extract with aqueous extract in Section 11.5.7.

11.5. Water Sample Extraction

11.5.1. Allow the sample to reach ambient temperature. Weigh and record the bottle for determination of initial volume. Add 1 mL of the internal standard solution diluted in acetone (See Table 2) to the sample bottle. Cap the bottle and mix the sample by gently shaking for 30 seconds. Filter the sample through a glass fiber filter that has been rinsed with toluene. Reagent water used as a blank must also be filtered in a similar fashion and subjected to the same cleanup analysis as the water samples.

11.5.1.1. If the total dissolved and suspended solids contents are too much to filter through the glass fiber filter, centrifuge the sample, decant, and then filter the aqueous phase. Combine the solids from the centrifuge bottle(s) with the particulate on the filter into an extraction thimble and proceed to Section 11.4.2.1.

11.5.2. The filtered aqueous sample is poured into a 2 L separatory funnel. Add 100 mL methylene chloride to the sample bottle, seal, and shake for 60 seconds to rinse the inner surface.

11.5.3. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst shall employ mechanical techniques to complete the phase separation (i.e. glass stirring rod) or chemical separation (i.e. introduction of dilute acid or base, consult supervisor prior to proceeding with this procedure).

Warning: The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people or other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed.

11.5.4. Drain the methylene chloride extract into a 500 mL round bottom flask by passing the extract through a funnel packed with a glass wool plug and half-filled with anhydrous sodium sulfate.

- 11.5.5. Extract the water sample two more times using 100 mL of fresh methylene chloride each time. Drain each extract through the funnel into a round bottom flask. After the third extraction, rinse the sodium sulfate with at least 30 mL of fresh methylene chloride. Concentrate this extract as described in Section 11.6.

Note: The analyst may also drain the entire extract from the separatory funnel back into the 1 L amber glass jar. Then filter the entire 300 mL through a funnel containing a glass wool plug and half-filled with anhydrous sodium sulfate (glass wool and sodium sulfate are pre-rinsed with methylene chloride prior to use) followed by a final methylene chloride rinse then proceeding to the concentration step described in Section 11.6.

- 11.5.6. Determine the original sample volume by weighing the empty sample container and assume the specific gravity of the sample to be 1.0 g/mL. Record the sample volume.

- 11.5.7. Combine the aqueous extract with the filter extract and again concentrate using Section 11.6.

Note: The combination of the aqueous extract and filter extract can be performed in any way possible ensuring proper combination of extracts.

11.6. Macro-Concentration Procedures (All Matrices)

- 11.6.1. Prior to cleanup, extracts from all matrices must be concentrated to approximately 2 mL (near dryness). In addition, the concentrated extracts from the aqueous filtrate and the filtered particulates must be combined prior to cleanup. Prior to concentrating all extracts add approximately 50 - 100 μ L of tetradecane.

11.6.2. Concentration by Rotary Evaporator

- 11.6.2.1. According to the manufacturer's instructions, assemble the rotary evaporator and warm the water bath to 65°C for toluene and 55°C for methylene chloride. Between samples, rinse 2 to 3 mL aliquots of toluene down the feed tube into a waste beaker.

- 11.6.2.2. Attach the round bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system and begin rotating the sample flask. Vacuum is not necessary to concentrate methylene chloride.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

- 11.6.2.3. Lower the flask into the water bath and adjust the speed of rotation and the temperature as required to complete the concentration in 10-20 minutes. At the proper rate of concentration, the flow of solvent into the receiving flask should be steady and no bumping or visible boiling of the extract shall occur.

NOTE: If the rate of concentration is too fast, analyte loss may occur.

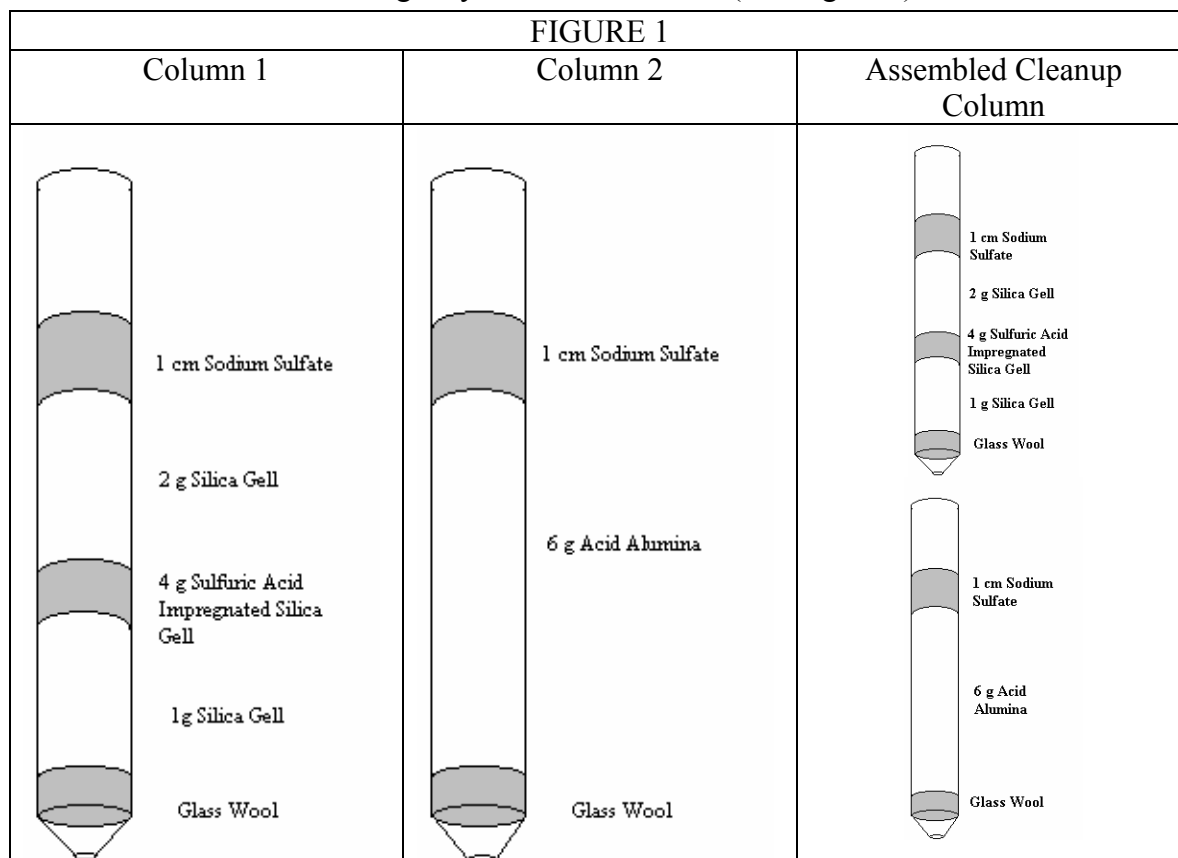
- 11.6.2.4. When the liquid in the concentration flask has reached an apparent volume of less than 2 mL, remove the flask from the water bath, and stop the rotation. Slowly and carefully emit air into the system. Be sure not to open the valve so quickly that the sample is blown out of the flask. Detach the flask from the rotary evaporator. Rinse the feed tube with approximately 2 mL of toluene and hexane prior to attaching the next extract.

11.7. Extract Cleanup Procedures (All Matrices)

- 11.7.1. Cleanup may not be necessary for every matrix. If particular circumstances require the use of a cleanup procedure, the analyst may use any or all of the procedures below or any other appropriate procedure. Before using a cleanup procedure, the analyst must demonstrate that the cleanup procedure accurately quantitates native amounts with minimal I.S. and CRS loss during the procedure. This can be achieved by processing 4 LCS replicates through the cleanup procedure.
- 11.7.2. Prior to cleanup, all extracts are spiked with the ^{37}Cl -2,3,7,8-TCDD cleanup standard. The recovery of this standard is used to monitor the efficiency of the cleanup procedures. Spike 50 μL of the cleanup standard solution (or a larger volume of diluted solution containing 12.5 ng of ^{37}Cl -2,3,7,8-TCDD) into each separatory funnel containing an extract, resulting in a concentration of 0.50 ng/ μL in the final extract analyzed by GC/MS.
- 11.7.3. Option C
- 11.7.3.1. Add 150 mL of hexane to the extract in the separatory funnel. Partition the concentrated extract against 40 mL of concentrated sulfuric acid. Shake for thirty seconds. Remove and discard the acid layer (bottom). Repeat the acid washing until there is no visible color in the acid layer. (Perform acid washings a maximum of four times).

WARNING: Concentrated sulfuric acid is hazardous and should be handled with care. A faceshield shall be worn when handling bulk acids and bases.

- 11.7.3.2. Partition the concentrated extract against 40 mL of distilled water. Shake for thirty seconds. Remove and discard the aqueous layer (bottom).
- 11.7.3.3. Dry the organic layer by pouring it through a funnel containing anhydrous sodium sulfate. Collect the extract in an appropriate size round bottom flask. Wash the separatory funnel with two 15 mL portions of hexane, pour through the funnel and combine the extracts. Concentrate the extracts to less than 2.0 mLs using the procedures described in Section 11.6.
- 11.7.4. Silica Gel and Alumina Column Chromatographic Procedure
- 11.7.4.1. Column 1: Insert a glass wool plug onto the bottom of a 20 mm disposable glass column. Add 1 g silica gel and tap the column gently to settle the silica gel. Add 4 g sulfuric acid-impregnated silica gel (may use a larger amount if necessary), and 2 g silica gel. Top with approximately 1 g anhydrous sodium sulfate. Tap the column gently after each addition (See Figure 1).



11.7.4.2. Column 2: Insert a glass wool plug onto the bottom of a 16 mm disposable glass column. Add 6 g of the activated acid alumina. Top with 1 g anhydrous sodium sulfate. Tap the top of the column gently (See figure 1).

11.7.4.2.1. Check each new batch of silica gel and alumina and maintain the results of the analyses on file. To accomplish this, combine an appropriate amount of the internal standard solution and an appropriate amount of matrix spike solution. Process this solution through both columns in the same manner as a sample extract (Section 11.7.4). Concentrate the QC sample to a final volume of 50 μ L. Proceed to analysis. If the recovery of any of the analytes is less than 80%, this batch of silica and/or alumina may not be used.

11.7.4.3. Add 20 mL of hexane to each column until the packing is free of air bubbles. A small positive pressure (5 psi) of clean dry nitrogen may be used if needed. Check the columns for channeling. If channeling is present, discard the column.

CAUTION: Do not tap a wetted column.

11.7.4.4. Assemble the two columns such that the eluate from Column 1 (silica gel) drains directly into Column 2 (alumina) (See Figure 1).

11.7.4.5. Transfer the extract from Section 11.6 to the top of the silica gel column. Rinse the flask with enough hexane (1 mL-2 mL) to complete the quantitative transfer of the sample to the surface of the silica and repeat at least one extra time.

11.7.4.6. Using 60 mL of hexane, elute the extract from Column 1 directly onto Column 2 which contains the alumina.

CAUTION: Do not allow the alumina column to run dry for a long period of time.

11.7.4.7. Add 10 mL of 20% methylene chloride/80% hexane (v/v) to Column 2, and elute until the solvent level is just below the top of the alumina.

11.7.4.8. Add 30 mL of 65% methylene chloride/35% hexane (v/v) to Column 2, and collect the eluate in a culture tube.

NOTE: Solvent percentages and solvent volumes may change due to change in packing material.

11.7.4.9. If the sample will require a carbon cleanup column, then add 200 μ L of tetradecane and concentrate the extract to near dryness using a turbo-evaporator apparatus. The extract is now ready to be

transferred to the carbon column. Otherwise, proceed to Section 11.8.

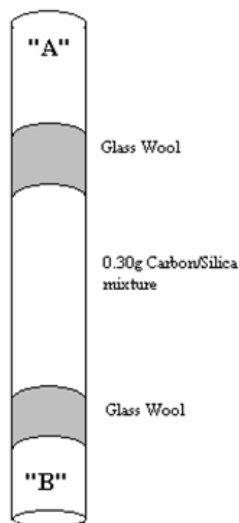
11.7.5. Carbon Column Chromatographic Procedure

11.7.5.1. Thoroughly mix 95 g activated silica gel and 5.0 g charcoal to yield a 5% w/w charcoal on silica (other weights may be used to yield the 5% w/w charcoal on silica). Activate the mixture at 190°C for twelve hours and store in a charcoal/silica oven at 190°C.

11.7.5.1.1. Check each new batch of the charcoal/silica and maintain the results from the analyses for examination. To accomplish this, add the appropriate amount of the internal standard solution and the appropriate amount of matrix spike solution to the cleanup column. Process the spiked solution in the same manner as a sample extract (Section 11.7.5). Concentrate the solution to 50 μ L and proceed with Section 11.8.3. If the recovery of any of the analytes is less than 80%, this batch of carbon/silica mixture may not be used.

11.7.5.2. Prepare an 8-inch glass column by cutting off each end of a 5 mL disposable serological pipette.

Figure 2 (Direction B Shown)



Note: A pre-cut column of similar diameter made by a glass manufacturer may also be used for this column. Insert a glass wool plug at one end of the column and pack it with 0.30 g of the charcoal/silica mixture. Insert an additional glass wool plug in the other end. See Figure 2.

CAUTION: It is very important that the column be packed properly to ensure that carbon fines are not carried into the eluate. PCDDs/PCDFs will adhere to the carbon fines and greatly reduce recovery. If carbon fines are carried into the eluate, filter the eluate using a 0.45 micron filter (pre-rinsed with toluene), then proceed to Section 11.8.

11.7.5.3. Pre-rinse with 5 mL hexane in both directions.

11.7.5.4. While the column is still wet, transfer the concentrated eluate from Section 11.6 to the prepared carbon column. Rinse the eluate container with two 1.0 mL portions of hexane and transfer the rinses to the carbon column. Elute the column with the following sequence of solvents:

11.7.5.4.1. Elute column with 2 mL of cyclohexane/methylene chloride (50:50 v/v).

11.7.5.4.2. Elute column with 2 mL of methylene chloride/methanol (75:25 v/v)

NOTE: The above two eluates may be collected, combined and used as a check on column efficiency.

11.7.5.5. Once the solvents have eluted through the column, turn the column over to the A direction and elute the PCDD/PCDF fraction with a minimum of 15 mL of toluene, and collect the eluate.

11.8. Final Concentration

11.8.1. Evaporate the 65% methylene chloride /hexane fraction from Section 11.7.4.8 or the toluene fraction from Section 11.7.5.5 to approximately 1.0 mL with N₂ via turbo-evaporator.

11.8.2. To a 2.0 mL conical vial (or equivalent) containing 50 µL of tetradecane and an appropriate amount of recovery standard solution (See Table 2), add the extract using small portions (~500 µL each rinse) of hexane rinses. Concentrate the sample using a concentration manifold with purified nitrogen. Transfer the 50 µL extract to an auto inject vial and transfer the sample label.

Note: a smaller final volume may be used to achieve a lower detection limit or CRQL. Client, project manager, and appropriate managers must be contacted and notified of such changes.

11.8.3. Submit sample for GC/MS analysis.

12. CALCULATIONS/DATA REDUCTION

Not applicable

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. The use of Roto-vaps and Turbo-vaps rather than Kuderna-Danish reduction allows extraction solvents to be collected and disposed of rather than released to the atmosphere.

- 14.2. The use of SoxTherm extraction rather than soxhlet extraction, when appropriate, reduces the volume of solvent used.
- 14.3. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.
- 14.4. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.
- 14.5. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.
- 14.6. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

- 15.1. Waste methylene chloride from glassware rinsing, glass fiber and sodium sulfate pre-rinsing, and extract rotary evaporator concentration. Keep waste methylene chloride separated from waste flammable solvents. Collect the waste methylene chloride in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel methylene chloride drum in the H3 closet. When the drum is full to no more than six and no less than four inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.
- 15.2. Waste acetone, hexane, toluene and other flammable solvents from glassware and acid rinsing, glassware pre-extraction and extract rotary evaporator concentration. Keep waste flammable solvents separated from waste methylene chloride. Collect the waste flammable solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel flammable solvent drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.
- 15.3. Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials that may or may not be contaminated/hazardous. Place contaminated materials into a contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the steel

collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

- 15.4. Various paper funnel filters, thimbles, glass wool, and extracted solids contaminated with methylene chloride or toluene. Dump the extracted solids, used sodium sulfate, glass wool, thimbles and filter paper into a contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.
- 15.5. Contaminated sulfuric acid used during extract cleanup. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.
- 15.6. Contaminated distilled water used during extract cleanup. Collect the contaminated water in a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the plastic drum to the waste collection area for shipment.
- 15.7. Extracted aqueous samples, contaminated with methylene chloride. Extracted aqueous samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.
- 15.8. Silica gel, alumina, carbon and sodium sulfate, from column clean-ups and extract drying, contaminated with various solvents and eluates. Put the contaminated materials into a contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

- 16.1. Corporate Environmental Health and Safety Manual.
- 16.2. Document Number DFLM 01.0, including Revision DFLM 01.1 (September 1991).
- 16.3. Update of Toxicity of Equivalency Factors (TEFs) for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-dioxins and Dibenzofurans (CDDs/CDFs), EPA 625/3-89/016 (March 1989).

16.4. SW-846, Method 8280A, Update III, December 1996.

16.5. Method 8280B, Revision 2, February, 2007

17. METHOD MODIFICATIONS

Unless otherwise specified, the modifications below apply to both method 8280A and method 8280B.

17.1. A 50 µL aliquot is no longer taken prior to addition of the recovery standard. This step was eliminated as the archived portion was never used and if a dilution of that manner is necessary, the sample is re-extracted at a smaller sample size.

17.2. Tridecane was replaced with tetradecane.

17.3. The stock solution concentrations and volumes added to extracts were adjusted for ease of application.

17.4. Silica and quartz sand is no longer necessary for extraction of solid matrices in the Soxhlet thimbles based on research of high resolution PCDF/PCDD analysis. Quartz sand may be used in samples if needed, such as for South Carolina samples, and is required in QC samples such as solid method blanks and laboratory control samples.

17.5. Method ASTM D2216 is now the universal percent water analysis for TestAmerica West Sacramento.

17.6. All cleanups are optional based on chemist judgement.

17.7. The amount of sulfuric acid-impregnated silica gel may be adjusted based on sample color or matrix.

17.8. The amount of solvent or the percentage of solvents used in the elution of the silica gel and alumina column or the carbon/silica column may be adjusted based on column profiles. (Based on current silica/alumina column profiles, 60 mL of hexane and 30 mL of 65% methylene chloride/hexane (v/v) is used instead of 90 mL of hexane and 26 mL of 80% methylene chloride /hexane (v/v).

17.9. NaCl and KOH were replaced with deionized water and NaOH for the acid-base washing procedure. The NaOH and second deionized water step is optional based on analyst judgement. Consult with area supervisor to determine if this is necessary for a particular sample.

18. ATTACHMENTS

18.1. Table 1- Matrix Spike Solution

18.2. Table 2 – IS, Cleanup Recovery, and Recovery Standard Solutions

19. REVISION HISTORY

19.1. WS-IDP-0011, Revision 2, Effective 09/30/2008

19.1.1. Updated to TestAmerica format.

19.1.2. Added changes to implement Method 8280B.

19.1.3. Editorial changes.

19.2. WS-IDP-0011, Revision 1.1, Effective 4/16/2008

19.2.1. Added new Sections 11.3.2.1 and 11.3.2.2 for using sand in South Carolina samples.

19.2.2. Clarified Sections 9.3 and 17.1.4 for South Carolina samples.

19.2.3. The preparation portion of the method was removed from SAC-ID-0011 into a separate SOP.

Table 1 Matrix Spiking Solution	
Analyte	Concentration (ng/μL)
2,3,7,8-TCDD	0.5
2,3,7,8-TCDF	0.5
1,2,3,7,8-PeCDF	1.25
1,2,3,7,8-PeCDD	1.25
1,2,3,6,7,8-HxCDF	1.25
1,2,3,6,7,8-HxCDD	1.25
1,2,3,4,6,7,8-HpCDF	1.25
1,2,3,4,6,7,8-HpCDD	1.25
OCDD	2.5
OCDF	2.5

This solution is prepared in isooctane (or nonane) and diluted with acetone prior to use. Only the ten isomers listed above will be used for evaluation. All seventeen 2,3,7,8-substituted isomers are incorporated into the matrix spike solution.

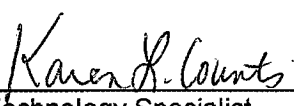
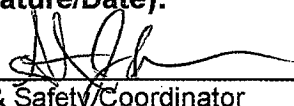


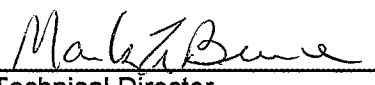
Table 2 Internal Standard, Recovery Standard, and Cleanup Standard Solutions			
Internal Standards	Stock Solution Concentration (ng/L)	Vol. Added to Sample (μL)	Final Amount (ng)
¹³ C-2,3,7,8-TCDD	0.5	50	25
¹³ C-2,3,7,8-TCDF	0.5	50	25
¹³ C-1,2,3,7,8-PeCDF ⁽¹⁾	0.5	50	25
¹³ C-2,3,4,7,8-PeCDF ⁽¹⁾	0.5	50	25
¹³ C-1,2,3,7,8-PeCDD ⁽¹⁾	0.5	50	25
¹³ C-1,2,3,4,7,8-HxCDF ⁽¹⁾	0.5	50	25
¹³ C-1,2,3,6,7,8-HxCDF ⁽¹⁾	0.5	50	25
¹³ C-2,3,4,6,7,8-HxCDF ⁽¹⁾	0.5	50	25
¹³ C-1,2,3,7,8,9-HxCDF ⁽¹⁾	0.5	50	25
¹³ C-1,2,3,4,7,8-HxCDD ⁽¹⁾	0.5	50	25
¹³ C-1,2,3,6,7,8-HxCDD	0.5	50	25
¹³ C-1,2,3,4,6,7,8-HpCDF	0.5	50	25
¹³ C-1,2,3,4,7,8,9-HpCDF ⁽¹⁾	0.5	50	25
¹³ C-1,2,3,4,6,7,8-HpCDD ⁽¹⁾	0.5	50	25
¹³ C-OCDD	1.0	50	50

(1) These extra internal standards are not mentioned in Method 8280A. These may be used to improve the method ruggedness and if interferences from these in other ions are not seen or significant.

The Internal Standard Solution and the Cleanup Standard Solution are prepared in isooctane. The Recovery Standard Solution may be prepared in either toluene or tetradecane.

**Title: INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION
SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT
ANALYSES**

[Method: SW846 Methods 6010A, 6010B and EPA Method 200.7]

Approvals (Signature/Date):			
	1/5/09		1/06/09
Technology Specialist	Date	Health & Safety Coordinator	Date
	1/5/09		1/5/09
Quality Assurance Manager	Date	Laboratory Director	Date
	1/5/09		
Technical Director	Date		

This SOP was previously identified as SOPNC-MT-012, Rev 0, dated 01/18/08

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1. SCOPE AND APPLICATION

- 1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Methods 6010A, 6010B and EPA Method 200.7. Tables I and IA of Appendix A lists the elements appropriate for analysis by Methods 6010A, 6010B and 200.7 and the associated reporting limit.. Additional elements may be analyzed under Methods 6010A, 6010B and 200.7 provided that the method performance criteria presented in Section 13.0 are met.
- 1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity, and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.
- 1.3. Methods 6010A and 6010B are applicable to the determination of dissolved, suspended, total recoverable, and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, biological, and TCLP, EP, and other leachates/extracts. All matrices require digestion prior to analysis. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.
- 1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis. Silver concentrations must be below 0.1 mg/L in aqueous samples.

2. SUMMARY OF METHOD

- 2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by radio frequency inductively-coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in

background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences must also be recognized, and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

- 2.1. Refer to NC-IP-010, Acid Digestion of Soils, SW846 Method 3050B, and NC-IP-011, Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods for details on sample preparation methods.

3. DEFINITIONS

- 3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).
- 3.2. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.
- 3.3. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.
- 3.4. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version for additional definitions. Refer to Appendix B for a cross reference of method definitions.

4. INTERFERENCES

- 4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:
 - Overlap of a spectral line from another element.
 - Unresolved overlap of molecular band spectra.
 - Background contribution from continuous or recombination phenomena.
 - Stray light from the line emission of high concentration elements.
- 4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.
- 4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the

sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

- 4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects) at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard, and/or use of a high solids nebulizer can reduce the effect. Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not significant with the ICP technique, but if observed can be minimized by buffering the sample, matrix matching, or standard addition procedures.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document.
- 5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.
- 5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
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Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4-ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3.1. The plasma emits strong UV light and is harmful to vision. **NOTE: AVOID looking directly at the plasma.**

5.3.2. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers must not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**. All samples with stickers that read “Caution/Use Hood!” **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Metals digestates can be processed outside of a fume hood. Solvent and waste containers must be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator

- 6.3. Argon gas supply, welding grade or equivalent
- 6.4. Coolflow or appropriate water cooling device
- 6.5. Peristaltic Pump
- 6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes – ranging from 5 μ L \rightarrow 10 ml
- 6.7. Class A volumetric flasks – range from 50 ml \rightarrow 2000 ml
- 6.8. Autosampler tubes – range from 8 ml \rightarrow 14 ml

7. REAGENTS AND STANDARDS

- 7.1 Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met. Additional information can be found in SOP NC-QA-017.
- 7.2 Working calibration, calibration verification solutions, and internal standard solutions must be prepared in a matrix of 5% hydrochloric and 5% nitric acids. Refer to Tables II, III, IV and V (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction, and spiking solutions. Refer to the laboratory Standard Logbook or Reagent Logbook for details on standard or reagent preparation.
- 7.3 Concentrated nitric acid (HNO_3), trace metal grade or better.
- 7.4 Concentrated hydrochloric acid (HCl), trace metal grade or better.
- 7.5 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Sample holding times for metals are six months from time of collection to the time of analysis.
- 8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in

either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

- 8.3. Soil samples do not require preservation, but must be stored at $4^{\circ}\text{C} \pm 2^{\circ}$ until the time of preparation.

9. QUALITY CONTROL

Table VI (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.1. Initial Demonstration of Capability

- 9.1.1. Prior to analysis of any analyte using Methods 200.7, 6010A, or 6010B, the following requirements must be met.
- 9.1.2. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used for each instrument. The IDL must be determined annually for client-specific projects. For DoD work, refer to SOP NC-QA-016. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. The IDL will be determined by multiplying by 3, the standard deviation obtained from the analysis of a blank solution, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples).
- 9.1.3. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. Refer to TestAmerica North Canton SOP NC-QA-021 and CA-Q-S-006 for details on MDL analysis and criteria.
- 9.1.4. Linear Range Verification (LR) - The linear range must be verified every six months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to verify the linear range limit must be analyzed during a routine analytical run, and must read within 10% of the expected value.

For the initial determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard must be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

- 9.1.5. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference, or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.
- 9.1.6. Inter-element Corrections (IECs) - ICP inter-element correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC, then the possibility of contamination must be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., ICP-MS). Published wavelength tables (e.g., MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than \pm the RL as defined in Tables I or IA. For elements with a reporting limit of 10 ug/L or less, the signal must be \pm two times the RL. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the actual concentration of the "interfering element."
- Note:** Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response. Additional spectral interference is present from easily ionizable elements such as potassium and sodium in axial viewing instruments.
- 9.1.7. Rinse Time Determination - Rinse times must be determined upon initial set-up of an ICP instrument. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see Section 9.1.4) must be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte

signals to < RL will define the rinse time for a particular ICP system. For some analytes, it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be re-analyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

- 9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level). For Ohio VAP projects, all analytes must be less than the reporting limit unless the associated samples are ND.
- If the analyte is a common laboratory contaminant (copper, iron, lead, or zinc), the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. Such action must be addressed in the project narrative.
 - Repreparation and re-analysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).
 - If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be addressed in the project narrative.
 - If the above criteria are not met and re-analysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative.
- 9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table II (Appendix A). The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the

laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits, the system is out of control and corrective action must occur. Unless in-house control limits are established, a control limit of 80 - 120% recovery must be applied.
- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The LCSD recovery is evaluated using the same control limits as the LCS. The RPD of the LCS and LCSD must be compared to in-house limits.
- In the instance where the LCS recovery is greater than the upper control limit and the sample results are < RL, the data may be reported with qualifiers. Such action must be addressed in the report narrative.
- Corrective action will be re-preparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP projects the corrective action must be re-preparation and reanalysis of the batch.

9.4 Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables II and V (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For Methods 200.7, 6010A, and 6010B, control limits of 75-125% recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include re-preparation and re-analysis of the batch. MS/MSD results, which fall outside the control limits, must be addressed in the narrative.
- If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data is reported as NC, MSB (i.e., not

calculated). Two other narrative notes for metals analyses: Matrix spike/spike duplicate spike recovery/recoveries was/were outside the acceptance limits of some analytes. The acceptable LCS analysis data indicated that the analytical system was operating within control and this condition is most likely due to matrix interference. See the Matrix Spike Report for the affected analytes which will be flagged with N. Matrix spike/spike duplicate relative percent difference (RPD) exceeded the acceptance limits for some analytes. The imprecision may be attributed to sample heterogeneity. See the Matrix Spike Report for the affected analytes, which will be flagged with *.

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.6 Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5 times (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample after correction for dilution must agree within 10% of the original sample determination when the original sample concentration is greater than 50 times the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists and the data is flagged.

9.12 Control Limits

9.12.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.12.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS (QC Browser program).

9.13 Method Detection Limits (MDLs) and MDL Checks

9.13.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.13.2 MDLs are easily accessible via LIMS (QC Browser program)

9.14 Nonconformance and Corrective Action

- 9.14.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the instructions in Appendix F.
- 10.3 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to Appendix F for detailed setup and operation protocols. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data
- 10.4 Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall within 10% of the true value for that solution. For Method 6010B, the relative standard deviation must be <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within \pm the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified (see Sections 11.6 through 11.8 for required run sequence).
- 10.5 Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the sample run. The CCV is to be a mid-range standard made from a dilution of the calibration standard. The CCV for all methods must fall within 10% of the true value for that solution. For Methods 6010B, and 200.7, the relative standard deviation must be <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV (see Sections 11.6 through 11.8 for required run sequence). The CCB result must fall within \pm RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, all affected samples will be re-analyzed with valid CCV/CCB pairs (refer to Section 11.7 for an illustration of the appropriate rerun sequence). Exceptions: If CCB > RL, samples < RL can be reported with an NCM. If CCV is outside of criteria on the high side, samples < RL can be reported with an NCM.

- 10.6 Verification Standards (6010A only). The calibration standards are analyzed against the calibration curve and must be within 5% of the true value.
- 10.7 Interference Check Analysis (ICSA/ICSAB) - The validity of the inter-element correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table IV (Appendix A) for the details of ICSA and ICSAB composition. Custom multi-element ICS solutions must be used. All analytes must be spiked into the ICSAB solution; therefore, if a non-routine analyte is required, then it must be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number, then the non-routine elements can be controlled from the ICSA as described in Section 10.6.3. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium, and magnesium must always be included in all ICP runs.
- 10.7.1 The ICSA and ICSAB solutions must be run at the beginning of the run (see Sections 11.6 or 11.7 for required run sequence). For Method 6010A, the ICSA and ICSAB solutions must be run every 8 hours.
- 10.7.2 The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.
- 10.7.3 ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within ± 2 times the RL from zero. ICSA results for the non-interfering elements with RLs > 10 ug/L must fall within ± 1 times the RL from zero. If the ICSA results for the non-interfering elements do not fall within \pm two times RL (RL ≤ 10) or $\pm 1 \times$ RL (RL > 10) from zero, the field sample data must be evaluated as follows.
- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
 - If the affected element was not required, then the sample data can be accepted.
 - If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than \pm two times the RL from zero, then the field sample data can be accepted.
 - If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than \pm two times the RL from

zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than ten times the analyte signal in the ICSA.

- If the data does not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples re-analyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually, the calculations must be clearly documented on the raw data.

10.8 CRI - To verify linearity near the RL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run. Additionally, some projects may require CRI analysis at the end of the run (see Sections 11.6 or 11.7 for required run sequence). Evaluate associated samples based upon advisory limits of $\pm 50\%$ of true value. This standard is required for Method 6010A.

Note: The custom CRI mix contains most analytes at a level near the standard lab reporting limit.

11. PROCEDURE

- 11.1. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.
- 11.2. Prior to calibration and between each sample/standard, the system is rinsed with the calibration blank solution.
- 11.3. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB), and field samples (linear range) to improve the data review process.
- 11.4. To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.
- 11.5. The following procedural guidelines must be followed when using an internal standard:
 - 11.5.1 Typically used internal standard is: yttrium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)
 - 11.5.2 The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards must be added to blanks, samples, and

standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

- 11.5.3 The concentration of the internal standard must be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.
- 11.5.4 The internal standard raw intensity counts must be printed on the raw data.
- 11.5.5 The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte). The instrument automatically adjusts sample results based on comparison of the internal standard intensity in the sample to the internal standard intensity at calibration.
- 11.5.5.1 If the internal standard counts fall within $\pm 30\%$ of the counts observed in the ICB, then the data is acceptable.
- 11.5.5.2 If the internal standard counts in the field samples are more than $\pm 30\%$ higher than the expected level, evaluate the run to determine if a dilution is needed due to matrix interference.

- 11.6 The following analytical sequence must be used for Methods 6010B, and 200.7:

Instrument Calibration

ICV

ICB

CRI

ICSA

ICSAB

CCV

CCB

10 samples

CCV

CCB

10 samples

CCV

CCB

Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run

CRI (The CRI counts as a sample analysis.)

CCV

CCB

Refer to Quality Control Section 9.0 and Table VI (Appendix A) for Methods 6010A,

6010B, and 200.7 quality control criteria.

- 11.7 The following analytical sequence must be used for Methods 6010A:

Instrument Calibration

ICV

ICB

CRI (+/- 50%)

Calibration verification standards

ICSA (maximum every 8 hours)

ICSAB (maximum every 8 hours)

CCV

CCB

10 samples

CCV

CCB

10 samples

CCV

CCB

Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run

CRI (The CRI counts as a sample analysis.)

CCV

CCB

- 11.8 The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 10.4.

Original Run: Instrument Calibration

ICV

ICB

CRI

ICSA

ICSAB

CCV

CCB

10 samples

CCV

CCB

10 samples

CCV

CCB

10 samples **

CCV *

* Failure occurs at CCV/CCB

CCB *

**Samples requiring rerun for affected analytes

10 samples **

CCV
CCB
10 samples
CCV
CCB

- 11.9 The instrument may be reprofiled between CCV/CCB pairs to correct for environment- induced drift.
- 11.10 Guidelines are provided in the Appendix C, D, and E on procedures to minimize contamination of samples and standards, preventive maintenance, and troubleshooting.
- 11.11 All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range. If an inter-element correction exists for an analyte, which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.
- 11.12 Any variation in procedure must be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report; and is approved by a Supervisor/Group Leader and QA Manager.
- 11.13 Nonconformance documentation must be filed in the project file.
- 11.14 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.
- 11.15 Analytical Documentation
 - 11.15.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.
 - 11.15.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.
 - 11.15.3 Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.
 - 11.15.4 Sample results and associated QC are entered into the LIMs after final

technical review.

12 DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100 \left(\frac{Found(ICV)}{True(ICV)} \right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \left(\frac{Found(CCV)}{True(CCV)} \right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \left(\frac{SSR - SR}{SA} \right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100 \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg / L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples is calculated as follows:

$$mg / Kg, dry weight = \frac{C \times V \times D}{W}$$

Where:

C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \left(\frac{Found(LCS)}{True(LCS)} \right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading \times 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Trivalent Chromium

12.10.1. Trivalent chromium (CR^{+3}) is the result obtained by subtracting the hexavalent chromium (CR^{+6}) results for a sample from the total chromium result from that sample. The total chromium result is determined using the procedures in this SOP. The hexavalent chromium result is determined using the procedures in TestAmerica North Canton SOP NC-WC-024.

12.10.2. Reporting Limits

12.10.2.1. The TestAmerica North Canton water reporting limit for trivalent chromium is 0.02 mg/l.

12.10.2.2. The TestAmerica North Canton solid reporting limit for trivalent chromium is 2.0 mg/kg, wet weight.

12.10.3. Calculations: $\text{Cr}^{+3} = \text{Cr, total} - \text{Cr}^{+6}$

13. METHOD PERFORMANCE

- 13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.
- 13.2. Refer to Tables I and IA in Appendix A for the list of analytes that may be analyzed using this SOP.
- 13.3. Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery must fall within $\pm 25\%$ and the MS/MSD must compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations greater than or equal to ten times the IDL. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples must recover within 20% of the true value or within the laboratory's historical acceptance limits.
- 13.4. Training Qualification
 - 13.4.1 The Group/Team Leader or the Supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

- 14.2. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the corporate environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

- 15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."
- 15.2. Waste Streams Produced by this Method

15.2.1. The following waste streams are produced when this method is carried out:

15.2.2. Acid waste consisting of sample and rinse solution - Any sample waste generated must be collected and disposed of in the acid waste drum located in the Metals Lab.

15.2.3. Standards must be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. References

16.1.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits

16.1.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, July 1992. Method 6010A.

16.1.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.1.4. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7

16.1.5. Inductively Coupled Plasma – Atomic Emission Spectrometric Method for Trace Element Analysis of water and wastes Method 200.7, 40 CFR – Chapter I – Part 136 – Appendix C. Electronic version dated September 30, 2002

16.1.6. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.7. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.8. Revision History

Historical File:		Revision 2.0: 10/27/97		Revision 0: 01/08/04 (NC-MT-012)
(formerly CORP-MT-0001NC)		Revision 2.1: 04/19/99		
		Revision 3.1: 10/04/00		
		Revision 3.2: 01/19/01		
		Revision 3.3: 12/05/01		

	Revision 3.4: 01/08/04	
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16.2. Associated SOPs and Policies, latest version

16.2.1. TestAmerica North Canton QC Program, QA-003

16.2.2. Statistical Evaluation of Data and Development of Control Charts,
NC-QA-018

16.2.3. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and
CA-Q-S-006

16.2.4. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.5. Hexavalent Chromium (Colorimetric), NC-WC-024

16.2.6. Acid Digestion of Soils, SW846 Method 3050B, NC-IP-010

16.2.7. Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series
Methods, NC-IP-011 Standards and Reagents, NC-QA-017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from Methods 6010B and 200.7.

17.1.1.1. TestAmerica North Canton Laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in-house as recommended by the subject methods.

17.1.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. In determining IECs because of lack of definition clarification for "concentration range around the calibration blank," TestAmerica North Canton has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.3. Whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical

interference exists. Because TestAmerica North Canton laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, TestAmerica North Canton may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At TestAmerica North Canton, matrix interference is determined by evaluating data for the LCS and MS/MSD. TestAmerica North Canton REQUIRES documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2. Modifications from Method 200.7

- 17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. TestAmerica North Canton labs utilize the IDL definition as defined in Section 9.1 of this SOP.
- 17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO₃/5% HCl instead of the specified 2% HNO₃/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.
- 17.1.2.3. Method Section 9.3.4 specifies that “Analysis of the ICV (ICSA/AB) solution immediately following calibration must verify that the instrument is within $\pm 5\%$ of calibration with a relative standard deviation $<3\%$ from replicate integrations ≥ 4 ”. TestAmerica North Canton uses a minimum of two exposures.
- 17.1.2.4. The 40 CFR version of Method 200.7 requires the instrument check standard to agree within $\pm 5\%$ of expected values. Also, the 40 CFR version requires the interference check sample to be analyzed at the beginning, end, and at periodic intervals throughout the sample run. At TestAmerica North Canton, the instrument check standard equals the CCV, which must agree within $\pm 10\%$ of expected values, and the ICSA standards are analyzed only at the beginning of a sample run. TestAmerica’s procedures are in line with the Rev. 4.4, May 1994 version of Method 200.7.
- 17.1.2.5. Section 7.12 of Method 200.7 indicates that the QCS (ICV) must be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations

which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

- 17.1.2.6. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 Section 10.4 states that results must fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.
- 17.1.2.7. Method 200.7 Section 9.3.4 states the CCB must be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica North Canton has adopted an absolute control limit of \pm RL from zero for calibration blank criteria. SOP Section 10.4 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B

- 17.1.3.1. Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.
- 17.1.3.2. Method 6010B Section 8.6.1.3 states that the results of the calibration blank are to agree within three times the IDL. If not, repeat the analysis two or more times, and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and re-analyze the previous ten samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica North Canton has adopted an absolute control limit of \pm RL from zero for calibration blank criteria. See SOP Section 10.4 for a detailed description of the required corrective action procedures.

17.1.4 Modifications from Method 6010A

- 17.1.4.1 Chapter 1 of SW846 states the method blank should not contain any analyte of interest at, or above, the MDL. This SOP states the method blank must not contain any analyte of interest at, or above, the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank.

- 17.1.4.2 Calibration is performed according to instrument manufacturer's recommendations. Method 6010A provides contradictory instructions for instrument calibration by stating to calibrate both according to instrument manufacturer's recommendations as well as to use a blank and three standards. TJA has stated that the use of multiple standards may be detrimental to determinations near the detection limit due to the inability of the linear equation to force-fit through the origin, and recommends the calibration be performed using a blank and one standard. This SOP requires verification of the initial instrument calibration using a CRI at two times the RL, an ICV at 5 - 25% of calibration level, a CCV at 50% of calibration level, and by rerunning the high calibration standard (HCAL) post calibration to demonstrate linearity (See Tables I and IA).
- 17.1.4.3 Section 5.6 of Method 6010A states the instrument check standard (CCV) should be prepared from a second source standard. This SOP states the CCV will be from the same source as the calibration standards. The purpose of the second source standard is to verify the accuracy of the calibration standards. The intent of this requirement is met through the analysis of a second source ICV standard prior to the analysis of samples. The use of a same source CCV provides for a more accurate and consistent measure of instrument drift from initial calibration.
- 17.1.4.4 Section 5.7 of Method 6010A states spiking of the ICS solution with analytes is not required if the ICP will display over-correction as a negative number. All TestAmerica instrumentation has this capability, and therefore, the spike analysis is not required. TestAmerica does run a spiked ICSAB, but the analytes are not spiked at the 10x IDL level referenced in Method 6010A. The ICSAB solution run by TestAmerica is based on the design of the ICSA/ICSAB solution provided by EPA directly to contract environmental labs.
- 17.1.4.5 Method 6010A uses a Quality Control Standard (QCS) on a weekly basis to verify calibration standard accuracy. TestAmerica refers to QCS as an ICV, and the accuracy verification is performed on a daily basis. The QCS described in Method 6010A is made to contain analytes at 10x the IDL. The TestAmerica ICV solution is not made at 10x IDL for all elements as this concentration is not appropriate relative to the standard reporting limits. TestAmerica designed the ICV to be a reliable indicator of calibration standard accuracy by raising the analyte concentrations to a level where the analytical determination is not impacted by low-level curve bias.

- 17.1.4.6 Method 6010A states the CCB should be within three times the standard deviations of the average blank reading. The intent of this requirement is to ensure the calibration is not drifting at the low end. In the absence of guidance on how to determine the mean blank level from EPA, TestAmerica has adopted an absolute control limit of \pm RL from zero for calibration blank criteria.

APPENDIX A

TABLES

TABLE I. Methods 200.7, 6010A, and 6010B Target Analyte List

Element	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	200	20
Antimony	Sb	7440-36-0	60	6
Arsenic	As	7440-38-2	300	30
Barium	Ba	7440-39-3	200	20
Beryllium	Be	7440-41-7	5.0	0.5
Boron	B	7440-42-8	200	20
Cadmium	Cd	7440-43-9	5.0	0.5
Calcium	Ca	7440-70-2	5000	500
Chromium	Cr	7440-47-3	10	1
Cobalt	Co	7440-48-4	50	5
Copper	Cu	7440-50-8	25	2.5
Iron	Fe	7439-89-6	100	10
Lead	Pb	7439-92-1	100	10
Magnesium	Mg	7439-95-4	5000	500
Manganese	Mn	7439-96-5	15	1.5
Molybdenum	Mo	7439-98-7	40	4
Nickel	Ni	7440-02-0	40	4
Potassium	K	7440-09-7	5000	500
Selenium	Se	7782-49-2	250	25
Silver	Ag	7440-22-4	10	1
Sodium	Na	7440-23-5	5000	500
Thallium	Tl	7440-28-0	2000	200
Vanadium	V	7440-62-2	50	5
Zinc	Zn	7440-66-6	20	2
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5

TABLE IA. Method 200.7, 6010A, and 6010B Trace ICP Target Analyte List

Element	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Concentration (ug/L)
Aluminum	2000
Antimony	500
Arsenic	2000
Barium	2000
Beryllium	50
Cadmium	50
Calcium	50000
Chromium	200
Cobalt	500
Copper	250
Iron	1000
Lead	500
Magnesium	50000
Manganese	500
Molybdenum	1000
Nickel	500
Potassium	50000
Selenium	2000
Silver	50
Sodium	50000
Thallium	2000
Vanadium	500
Zinc	500
Boron	1000
Tin	2000
Titanium	1000

TABLE III . Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	5	1000	2000
Cadmium	1000	2	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	100000	5000	25000	50000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	100000	5000	25000	50000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000
Boron	10000	200	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000

TABLE IV. Interference Check Sample Concentrations

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	1000
Vanadium	-	500
Zinc	-	1000
Tin	-	1000
Boron		1000
Titanium		1000

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VI. Summary of Quality Control Requirements

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met		Terminate analysis. Correct the problem. Prepare new standards. Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery RSD dupl. exp < 3% Method 6010A: 90 - 110 % recovery Method 6010B: 90 - 110 % recovery RSD dupl. exp < 5%	Terminate analysis. Correct the problem. Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within \pm RL from zero.	Terminate analysis. Correct the problem. Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B: 90 - 110 % recovery RSD dupl. exp < 5% Method 6010A: 90 - 110 % recovery	Terminate analysis. Correct the problem. Recalibrate and rerun all samples not bracketed by acceptable CCV. Samples < RL can be accepted if the CCV is outside on high side with NCM.
CCB	Immediately following each CCV	The result must be within \pm RL from zero	Terminate analysis. Correct the problem. Recalibrate and rerun all samples not bracketed by acceptable CCB. Samples < RL can be accepted if the CCB is outside on high side with NCM.
CRI	Beginning of every run	50-150% recovery (advisory) (required for 6010A)	Evaluate associated samples.
Calibration verification	After the CRI	+/- 5%	Recalibrate

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
(6010A only)			
ICSA	Beginning of every run (maximum every 8 hours for 6010A)	See Section 9.9.3	See Section 9.9.3
ICSAB	Immediately following each ICSA (maximum every 8 hours for 6010A)	Results must be within 80 - 120% recovery	See Section 9.9.2.
Dilution Test	One per prep batch	For samples > 50 times the IDL, dilutions must agree within 10%	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples	<p>The result must be less than or equal to the RL.</p> <p>Common lab contaminants may be accepted up to 2x the RL (see Section 9.2). (not applicable for Ohio VAP)</p> <p>Sample results greater than 20 times the blank concentration are acceptable. (not applicable for Ohio VAP)</p> <p>Samples for which the contaminant is < RL may not require redigestion or re- analysis (see Section 9.2).</p>	<p>Redigest and re-analyze samples.</p> <p>Note exceptions under criteria section.</p> <p>See Section 9.2 for additional requirements.</p>
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples	<p>Aqueous LCS must be within 80 - 120% recovery or in-house control limits.</p> <p>Samples for which the contaminant is < RL and the LCS results are > 120% may not</p>	<p>Terminate analysis. Correct the problem. Re-digest and re- analyze all samples associated with the LCS.</p>

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
		require redigestion or re-analysis (see Section 9.3)	
Matrix Spike	One per sample preparation batch of up to 20 samples	75 - 125 % recovery or in-house control limits	In the absence of client specific requirements, flag the data. No flag required if sample level is > 4 times the spike added.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD \leq 20%	See Corrective Action for Matrix Spike.

* See Sections 11.6, 11.7, and 11.8 for exact run sequence to be followed.

APPENDIX B CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW 6010A/6010B, AND TESTAMERICA NORTH CANTON SOP

EPA 200.7	SW 6010A/6010B	TestAmerica North Canton SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	N/A	N/A
Laboratory fortified blank (LFB)	N/A	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

APPENDIX C - TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber
Instrument Drift	Replace torch (Crack) Clean or replace nebulizer (blockage) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Reprofile
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage (back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX D - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept scrupulously clean.

Powdered Gloves must not be used in the Metals Lab since the powder contains silica and zinc as well as other metallic analytes. Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

APPENDIX E - PREVENTATIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, indicate the date, time and instrument number. Then identify the problem and corrective action in the Maintenance Log.

The following procedures are required to ensure that that the instrument is fully operational:

	Change sample pump tubing and pump windings
As Needed	Check rinse solution and fill if needed
	Check waste containers and empty if needed
	Check sample capillary tubing is clean and in good condition
	Check droplet size to verify nebulizer is not clogged.
	Check sample flow for cross flow nebulizer
	Check pressure for vacuum systems
	Clean plasma torch assembly to remove accumulated deposits
	Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance
	Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
	Apply silicon spray on autosampler tracks
	Check water level in cool flow
	Change oil for vacuum systems
	Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

APPENDIX F ICP Operating Instructions

ICP Analysis (TJA 61E) Example

1. SETUP

- a. Plasma Control Panel (enter)
- b. (F1)-Startup
- c. (F9)-Continue
- d. (F2)-Levels
 1. Change auxiliary gas to low – use space bar to toggle
 2. Change nebulizer gas flow to 0.5 L/min.
 3. Change pump rate to 130
 4. Esc
 5. Allow instrument to warm up approximately 30 minutes.

2. DEVELOPMENT

- a. Methods (enter)
- b. Enter method name
- c. (F3)-Method Info.
- d. Change file name
- e. (F9)-Done
- f. (F9)-Done/Keep

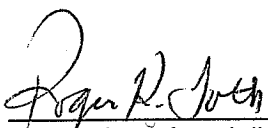
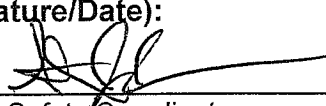



3. OPERATION

- a. Analysis (enter)
- b. (F5)-Profile
 1. (F3)-Automatic
 2. (F1)-Run

3. If peak position is greater than ± 0.05 units from the center peak position, you must adjust the profile. If it is within ± 0.05 units, press (F9)-done.
 4. To adjust select (F1)-CalcSS and enter current vernier position. (enter)
 5. Adjust to new vernier position (F9)-done
 6. Rerun profile until peak position is ± 0.05 units.
 7. (F9) - Done
- c. Autosampler (F9)
1. Enter method name (enter)
 2. Enter autosampler table name (enter)
 3. (F1) - Run

Title: ACID DIGESTION FOR AQUEOUS SAMPLES

[Methods: SW846 and MCAWW 200 Series]

Approvals (Signature/Date):			
	1-2-09		1/02/09
Technology Specialist	Date	Health & Safety Coordinator	Date
	1/5/09		1/2/09
Quality Assurance Manager	Date	Technical Director	Date
	1/5/09		
Laboratory Director	Date		

This SOP was previously identified as SOP No. CORP-IP-0003NC, Rev 1.6, dated 02/07/07

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1. **SCOPE AND APPLICATION**

- 1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) using the MCAWW 200 series methods (NPDES) and SW846 Methods 3005A, and 3010A.
- 1.2. The applicability of each of these preparation protocols to specific analytes is detailed in Tables I and II (Appendix A). Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.
- 1.3. This SOP provides procedures applicable to the preparation of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, certain aqueous sludges, and leachates/extracts.
- 1.4. SW-846 Method 3005A / MCAWW Method 200.8 are used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP and ICPMS.
- 1.5. MCAWW Method 200.7 is used to prepare surface water, domestic and industrial waste samples for total, total recoverable, and dissolved metals determination by ICP.
- 1.6. SW-846 Method 3010A is used to prepare aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.

2. **SUMMARY OF METHOD**

- 2.1. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP and ICPMS
 - 2.1.1 A representative aliquot of sample is heated with nitric and hydrochloric acids and reduced to a low volume. The digestate is filtered (if necessary) and diluted to volume.
- 2.2. Method 3010A / Method 200.7 / - Preparation for Total Metals Analysis by ICP
 - 2.2.1 A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary), and diluted to volume.

3. **DEFINITIONS**

Additional definitions of terms used in this SOP may be found in the glossary of the QAM.

- 3.1. Dissolved Metals: Those elements which pass through an 0.45 um membrane (sample is acidified after filtration).
- 3.2. Total Metals: The concentration determined on an unfiltered sample following digestion.
- 3.3. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot dilute mineral acid.

4. **INTERFERENCES**

- 4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.
- 4.2. The entire work area, including the bench top and fume hood, must be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix B for additional contamination control guidelines.
- 4.3. Boron from the glassware will migrate into the sample solution during and following sample processing. For critical low level determinations of boron, it is recommended quartz or plastic labware be used.
- 4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble with acids.
- 4.5. Visual interferences or anomalies (such as dilution due to oily matrix) must be documented.
- 4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric acid media.
- 4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.
- 4.8. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document.
- 5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.
- 5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

- 5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

- 5.5. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.
- 5.6. Exposure to chemicals must be maintained **as low as reasonably achievable**. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.
- 5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.
- 5.8. Always carry bulk concentrated acid bottles in appropriate impact proof containers.
- 5.9. Acid spills must be neutralized immediately, flushed with water, and cleaned up using appropriate spill kits.
- 5.10. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire-polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

- 6.1. Hot plate, digestion block, or other adjustable heating source capable of maintaining a temperature of 90-95°C
- 6.2. Calibrated thermometer that covers a temperature range of 0-200°C
- 6.3. Griffin beakers of assorted sizes or equivalent. Equivalent containers may be disposable digestion cups for digestion blocks which are certified by the manufacturer and calibrated by the laboratory.
- 6.4. Watch glasses, ribbed or equivalent
- 6.5. Whatman No. 41 filter paper or equivalent
- 6.6. Funnels or equivalent filtration apparatus
- 6.7. Graduated cylinder or equivalent capable of measuring 50 mL within 3% accuracy
- 6.8. Analytical balance capable of accurately weighing to the nearest 0.01 grams
- 6.9. Repeaters or suitable reagent dispensers

- 6.10. Calibrated automatic pipettes with corresponding pipette tips
- 6.11. Class A volumetric flasks
- 6.12. pH indicator strips (pH range 0 - 6)
- 6.13. Plastic digestate storage bottles, such as Corning Snap Seals (may be used if their accuracy is documented and is better than 2%)

7. REAGENTS AND STANDARDS

- 7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.
- 7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.
- 7.3. Working ICP LCS/MS spike solution: Prepare the ICP LCS/MS working spike solution from custom stock standards to the final concentration listed in Table III. The working spike must be prepared in a matrix of 5% HNO₃. This acid (5 mL of concentrated HNO₃ per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months.
- 7.4. The ICPMS LCS/MS spike solution is provided directly by the vendor. No further standard preparation is necessary.
- 7.5. The TCLP MS working spike solution is provided directly by the vendor, no further standard preparation is necessary. Refer to Table V for final digestate spike concentrations.
- 7.6. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

- 7.7. Aqueous laboratory control samples (LCS) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Tables III and IV (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP and ICPMS LCS and matrix spike preparations.
- 7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better
- 7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better
- 7.10. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid, a violent reaction may occur.

8. **SAMPLE COLLECTION, PRESERVATION AND STORAGE**

- 8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.
- 8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and must be stored in either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.
- 8.3. For dissolved metals analysis, the samples must be filtered through a 0.45 μm filter prior to preservation. Filtration must be done in the field. In the event that samples are not field filtered, filtration occurs in the laboratory prior to preparation.

9. **QUALITY CONTROL**

Table VI (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

- 9.1. Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs. In cases where there is insufficient sample volume to perform an MS/MSD, an LCS/LCS duplicate is required.
- 9.2. Sample Count: Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count for determining the size of a preparation batch.

- 9.3. Method Blank (MB): One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.
- 9.3.1 Aqueous method blanks are prepared by taking 50 mL of reagent water through the appropriate procedure as described in Section 11.
- 9.3.2 TCLP method blanks are prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.
- 9.4. Laboratory Control Sample (LCS): One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs. Refer to Sections 7.3 and 7.4 for instructions on preparation of the aqueous LCS spike solution.
- 9.4.1 The aqueous LCS is prepared by spiking a 50 mL aliquot of reagent water with 1.0 mL for ICP and 0.5 mL for ICPMS of the working LCS/MS spike solution (Sections 7.3 or 7.4). The LCS is then processed through the appropriate procedure as described in Section 11.
- Note: TCLP LCS is prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.
- 9.5. Additional information on QC samples can be found in QA Policy QA-003.
- 9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis.
- 9.6.1 The aqueous matrix spike sample is prepared by spiking a 50 mL aliquot of a sample with 1.0 mL for ICP and 0.5 mL for ICPMS of the working LCS/MS

spike solution (Sections 7.3 or 7.4). The matrix spike sample is then processed as described in Section 11.

- 9.6.2 The TCLP matrix spike sample is prepared by spiking a 50 mL aliquot of a leachate with 0.5 mL of the working TCLP spike solution (Section 7.5). The matrix spike sample is then processed as described in Section 11.

NOTE: The TCLP matrix spike must be added prior to preservation of the leachate.

NOTE: If analytes outside of the RCRA list are requested, 1 mL of additional spiking solution(s) are added.

- 9.6.3 If insufficient sample is available to process a MS/MSD, then a second LCS must be processed.

9.7. Control Limits

- 9.7.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

- 9.7.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS (QC Browser program).

9.8. Method Detection Limits (MDLs) and MDL Checks

- 9.8.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

- 9.8.2 MDLs are easily accessible via LIMS (QC Browser program)

9.9. Nonconformance and Corrective Action

- 9.9.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

- 10.1 The hotplate/hotblock temperature must be verified daily for each hotplate used, and must be recorded on a hotplate/hotblock temperature log.

11. **PROCEDURE**

- 11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file.
- 11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.
- 11.3. All digestion procedures must be carried out in a properly functioning hood.
- 11.4. All samples are to be checked out of Sample Control with an electronic chain of custody.
- 11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample.
- 11.6. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment, etc.), contact the lab supervisor or project manager for further instructions. In some cases, it may be more appropriate to process these samples as solids.
- 11.7. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and Reporting group.
- 11.8. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.
- 11.9. For DoD work, refer to SOP NC-QA-016 for specific details.
- 11.10. The following procedure must be followed for all aqueous sample preparations.
 - 11.10.1 Mix sample by shaking the container.
 - 11.10.2 Measure 50 mL of the sample into a calibrated digestion tube. (Beakers may be used for oil matrices.)

- 11.10.3 Measure two extra aliquots of sample selected for the MS/MSD analysis. Spike each aliquot with the appropriate spiking solutions (Sections 7.3 to 7.5 and 9.6).
- 11.10.4 Measure 50 mL of reagent water into a calibrated digestion tube for the method blank.
- 11.10.5 Measure 50 mL of reagent water into a calibrated digestion tube for the LCS and add the appropriate spiking solutions (Sections 7.3 to 7.5 and 9.6).
- 11.11. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP / ICPMS
 - 11.11.1 To the sample container, add 1 mL of concentrated HNO₃ and 2.5 mL of concentrated HCl.
 - 11.11.2 Cover with ribbed watch glass.
 - 11.11.3 Heat at 90-95°C until volume is reduced to between 15 and 20 mL. For DoD samples, the final volume is reduced to between 5 and 10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.
 - 11.11.4 Cool the beaker in a fume hood.
 - 11.11.5 Filter sample, if insoluble materials are present, through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.
 - 11.11.6 Rinse container and filter paper with reagent water to ensure complete sample transfer.
 - 11.11.7 Adjust the final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.
- 11.12. Method 3010A / Method 200.7 - Preparation for Total Metals Analysis by ICP Spectroscopy

- 11.12.1 To the sample container, add 3.0 mL of concentrated HNO₃.
- 11.12.2 Cover with ribbed watch glass.
- 11.12.3 Place container on hotblock 90-95°C, and evaporate to a volume of 15-20 mL, while ensuring that no portion of the bottom of the beaker is allowed to go dry. For DoD projects, the sample is evaporated to 5-10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

- 11.12.4 Add 5 mL of 1:1 HCl.
- 11.12.5 Cover and reflux for an additional 15 minutes to dissolve precipitate or residue. Cool in a fume hood.
- 11.12.6 Filter sample, if insoluble materials are present, through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™.

Note: If any samples in the QC batch are filtered, the method blank and LCS associated with that batch must also be filtered.

- 11.12.7 Rinse container and filter paper with reagent water to ensure complete sample transfer.
- 11.12.8 Adjust final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered, or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.

11.13. Analytical Documentation

- 11.13.1 Record all analytical information in the analytical logbook/logsheets, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.
- 11.13.2 All standards and reagents are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.
- 11.13.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

- 11.13.4 Sample results and associated QC are entered into LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

- 13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The spiking level should be equivalent to a mid-level calibration. (For certain tests more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

- 13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

- 13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

13.2. Training Qualification

- 13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

- 13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

- 14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. **WASTE MANAGEMENT**

15.1. All waste must be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention”.

15.2. Waste Streams Produced by the Method

15.2.1 The following waste streams are produced when this method is carried out.

15.2.1.1 Acidic waste containing nitric acid generated by the extraction. This waste is disposed of in the designated container labeled “Acid Waste”.

15.2.1.2 Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container identified as “Solid Waste”.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by an annual refresher training.

16. **REFERENCES**

16.1. References

16.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A and 3010A

16.1.2 Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983

16.1.3 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.4 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.5 Revision History

Historical File:		Revision 1.2: 03/20/00		Revision 1.6: 02/07/07
(formerly CORP-IP-0003NC)		Revision 1.3: 09/25/01		Revision 0: 09/30/08 (NC-IP-011)
		Revision 1.4: 02/19/03		
		Revision 1.5: 12/07/04		

16.2. Associated SOPs and Policies, current version

16.2.1 TestAmerica QC Program, QA-003

16.2.2 Glassware Washing, NC-QA-014

16.2.3 Statistical Evaluation of Data and Development of Control Charts,
NC-QA-018

16.2.4 Method Detection Limits and Instrument Detection Limits, CA-Q-S-006 and
NC-QA-021

16.2.5 Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6 Standards and Reagents, NC-QA-017

16.2.7 Subsampling, NC-IP-001

16.2.8 Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric
Method for Trace Element Analyses, SW846 Methods 6010A, 6010B, and
200.7, NC-MT-012

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference methods

17.1.1 Modifications applicable to SW-846 reference methods

17.1.1.1 The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "flexibility to alter digestion volumes is

addressed and “allowed” by Table 3-1 and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples. EMSL-Ci has also taken the stance that “reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology.” Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated “As a “representative sample” can be assured, scaling causes no loss of precision and accuracy in the analysis.”

17.1.2 Modifications Specific to Method 3010A

- 17.1.2.1 Section 11.11.3 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL, but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

17.1.3 Modifications Specific to MCAWW Methods

- 17.1.3.1 It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 11.0. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness vs. an exact volume).

APPENDIX A

TABLES

TABLE I. Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010 A
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	
Arsenic	As	7440-38-2	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element.

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE II. Approved Preparation Method Analytes – NPDES

ELEMENT	Symbol	CAS Number	200.7 (9.4)	200.7 (9.3)
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	X
Arsenic	As	7440-38-2	X	X
Boron	B	7440-42-8	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silicon	Si	7631-86-9	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE III. ICP Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/ MS Level * (ug/l)
Aluminum	100	2000
Antimony	25	500
Arsenic	100	2000
Barium	100	2000
Beryllium	2.5	50
Cadmium	2.5	50
Calcium	2500	50000
Chromium	10	200
Cobalt	25	500
Copper	12.5	250
Iron	50	1000
Lead	50	500
Magnesium	2500	50000
Manganese	25	500
Molybdenum	50	1000
Nickel	25	500
Potassium	2500	50000
Selenium	100	2000
Silver	2.5	50
Sodium	2500	50000
Thallium	100	2000
Vanadium	25	500
Zinc	25	500
Boron	50	1000
Tin	100	2000
Titanium	50	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.3) to 50 mL of sample.

TABLE IV. ICPMS Aqueous LCS and Matrix Spike Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)
Aluminum	1000	10000
Antimony	10	100
Arsenic	10	100
Barium	10	100
Beryllium	10	100
Cadmium	10	100
Calcium	1000	10000
Chromium	10	100
Cobalt	10	100
Copper	10	100
Iron	1000	10000
Lead	10	100
Magnesium	1000	10000
Manganese	10	100
Molybdenum	10	100
Nickel	10	100
Potassium	1000	10000
Selenium	10	100
Silver	10	100
Sodium	1000	10000
Strontium	10	100
Thallium	10	100
Vanadium	10	100
Zinc	10	100
Boron	10	100
Tin	10	100
Titanium	10	100

- * Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.5 mL working spike (Section 7.4) to 50 mL of sample.

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels for ICP

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)*
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.5 mL working spike (Section 7.5) to 50 mL of sample.

TABLE VI. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: - NC-MT-0002 - NC-MT-012	Redigest and re-analyze samples associated with the method blank.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: - NC-MT-0002 - NC-MT-012	Redigest and re-analyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: - NC-MT-0002 - NC-MT-012	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: - NC-MT-0002 - NC-MT-012	See Corrective Action for Matrix Spike.

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains zinc, as well as other metallic analytes. Only vinyl or nitrile gloves must be used in the metals laboratory.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

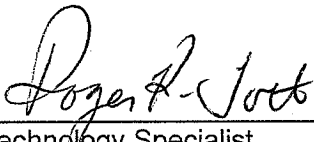




Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Title: ACID DIGESTION FOR SOIL SAMPLES

[Method: SW846 Method 3050B]

Approvals (Signature/Date):			
	1-2-09		1/02/09
Technology Specialist	Date	Health & Safety Coordinator	Date
	1/5/09		1/6/09
Quality Assurance Manager	Date	Laboratory Director	Date
	1/2/09		
Technical Director	Date		

This SOP was previously identified as SOP No. NC-IP-010, Rev 0, dated 07/18/08

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1. **SCOPE AND APPLICATION**

- 1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) as specified in SW846 Method 3050B.
- 1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICPMS for the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.
- 1.3. This method is not a total digestion, but will dissolve almost all metals that could become “environmentally available”. By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes, and sediments.
- 1.4. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. **SUMMARY OF METHOD**

- 2.1. A representative 1 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP and ICPMS analysis. The digestates are then filtered and diluted to 100 mL.

3. **DEFINITIONS**

- 3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), latest version.

4. **INTERFERENCES**

- 4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination, and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.
- 4.2. The entire work area, including the bench top and fume hood, must be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

- 4.3. Boron from the glassware may leach into the sample solution during, and following, sample processing. For critical low-level determinations of boron, only quartz and/or plastic labware are recommended.
- 4.4. Visual interferences or anomalies, such as foaming, emulsions, precipitates, etc., must be documented.
- 4.5. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric media.
- 4.6. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document.
- 5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.
- 5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.

Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

- 5.4 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.
- 5.5 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst must also be aware of the potential for a vigorous reaction.
- 5.6 Exposure to chemicals must be maintained **as low as reasonably achievable**. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.
- 5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.
- 5.8 Always carry bulk concentrated acid bottles in appropriate impact proof containers.
- 5.9 Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.
- 5.10 Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

- 6.1. Hot plate, digestion block, steam bath, or other heating source capable of maintaining a temperature of 91-99°C
- 6.2. Calibrated thermometer that covers a temperature range of 0-200°C
- 6.3. Vapor recovery device (Watch glasses, ribbed or other device)
- 6.4. Whatman No. 41 filter paper or equivalent
- 6.5. Funnels or equivalent filtration apparatus
- 6.6. Analytical balance capable of accurately weighing to the nearest 0.01 grams
- 6.7. Repeaters or suitable reagent dispensers
- 6.8. Calibrated automatic pipettes with corresponding pipette tips: 100uL, 500uL, 1mL-5mL
- 6.9. Class A volumetric flasks
- 6.10. Plastic digestate storage bottles, such as Corning Snap Seals™ (may be used if their accuracy is documented and is better than 2%)
- 6.11. Boiling Stones: Ultra Pure PTFE or equivalent

7. REAGENTS AND STANDARDS

- 7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.
- 7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.
- 7.3. Working ICP LCS/MS spike solution. Prepare the ICP LCS/MS working spike solutions from custom stock standards to the final concentration listed in Table II. The working spike must be prepared in a matrix of 5% HNO₃. This acid (5 mL of concentrated HNO₃ per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months. Refer to the Standards Logbook for details on standard preparation.

- 7.4. ICPMS LCS/MS spike solution. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are custom made so the final concentrations after spiking equals the concentrations listed in Table III.
- 7.5. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.
- 7.6. Nitric acid (HNO_3), concentrated, trace metal grade or better
- 7.7. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid a violent reaction may occur.
- 7.8. Hydrochloric acid (HCl), concentrated, trace metal grade or better
- 7.9. 30% Hydrogen peroxide (H_2O_2), Ultrapure grade

8. **SAMPLE COLLECTION, PRESERVATION, AND STORAGE**

- 8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of sample collection to the date of analysis.
- 8.2. Soil samples do not require preservation.

9. **QUALITY CONTROL**

Table IV (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 3050B the following requirements must be met.

- 9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The result of the MDL determination must be below the TestAmerica North Canton reporting limit. Criteria for DoD work is noted in SOP NC-QA-016.
- 9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The

results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOP NC-MT-012.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOPs. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.4.1. The MB is prepared by weighing a 1g aliquot of boiling chips. The MB is then processed as described in Section 11.9.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Table II provide the details regarding the stock, working standards, and final spike concentrations for ICP and ICPMS. Refer to Section 7.4 for instructions on preparation of the aqueous LCS.

9.5.1. The ICP LCS is prepared by spiking a 1g aliquot of boiling chips with 2 mL of the

working LCS/MS spike solution (Section 7.4). The ICPMS LCS is prepared by spiking a 1g aliquot of boiling chips with 1 mL of the LCS/MS solution (Section 7.4). The LCS is then processed as described in Section 11.9.

9.6. Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred. Table II provides the details regarding the stock, working standards and final matrix spike concentrations for ICP and ICPMS. Refer to Section 7.4 for instructions on preparation of the working matrix spike solutions.

9.7.1. The ICP matrix spike is prepared by spiking a 1g aliquot of sample with 2 mL of the working LCS/MS spike solution (Section 7.4) The ICPMS matrix spike is prepared by spiking a 1g aliquot of sample with 1 mL of the LCS/MS solution (Section 7.4) The matrix spike is then processed as described in Section 11.9.

10. CALIBRATION AND STANDARDIZATION

10.1. Hotblock temperature must be verified daily for each unit used, and must be recorded in a hotblock temperature log.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. The heating procedures are carried out in a properly functioning hood.

- 11.4. All samples are to be checked out of Sample Control with an electronic Chain-of-Custody.
- 11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. An automatic label printing program is used to reduce transcription errors (QuantIMS option).
- 11.6. Samples are typically logged in as either waters or soils. Wastes, such as organic liquids or sludges and tissues (animal/vegetable), are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation.
- 11.7. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and Reporting Group.
- 11.8. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards. Refer to Appendix B for details.
- 11.9. Preparation of Soils, Sediments, and Sludges for Analysis by ICP and ICPMS
 - 11.9.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.
 - 11.9.2. For each digestion procedure required (i.e., ICP or ICPMS), weigh a 1g portion of solid and record the exact weight to the nearest 0.01g. A 2g sample size may also be used if needed to meet the reporting limits.
 - 11.9.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.
 - 11.9.4. Add 10 mL of 1:1 HNO₃ and mix the sample.
 - 11.9.5. Heat sample to 95° ±4° C and reflux for 10 minutes without boiling, using a vapor recovery device.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be reprepared.
 - 11.9.6. Add 5 mL of concentrated HNO₃.
 - 11.9.7. Reflux at 95° ±4° C for 30 minutes. (Add reagent water, as needed, to ensure that the volume of solution is not reduced to less than 5 mL.)
 - 11.9.8. Add approximately 2 mL of reagent water and 1 mL of 30 % H₂O₂. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

- 11.9.9 Continue adding 30% H₂O₂ in 1 mL aliquots until effervescence is minimal or sample appearance is unchanged. Make sure effervescence subsides before each addition of H₂O₂.

Note: Do not add more than a total of 10 mL of 30 % H₂O₂.

- 11.9.10 Continue heating at 95° ± 4° C for two hours.

- 11.9.11 Add 10 mL of concentrated HCL and reflux for an additional 15 minutes without boiling.

- 11.9.12 Allow the sample to cool.

- 11.9.13 Filter sample through Whatman 41 filter paper or equivalent into a measuring bottle (for example, Corning Snap Seals™). These may be used if their accuracy is documented and is better than ± 2%. Rinse sample container and filter paper with reagent water to ensure complete sample transfer.

- 11.9.14 Dilute sample to 100 mL with reagent water. The sample is now ready for analysis.

11.10. Analytical Documentation

- 11.10.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

- 11.10.2. All standards and reagents are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

12. **DATA ANALYSIS AND CALCULATIONS**

Not applicable

13. **METHOD PERFORMANCE**

- 13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. Acceptance criteria are given in the determinative SOPs.

- 13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

- 13.3. Training Qualification

The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. **POLLUTION PREVENTION**

- 14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. **WASTE MANAGEMENT**

- 15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Acidic waste containing nitric acid generated by the extraction. This waste is disposed of in a designated container labeled "Acid Waste".

15.2.1.2. Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container labeled "Solid Waste".

16. **REFERENCES**

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996, Method 3050B

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.4. Revision History

Historical File:	Revision 2.1: 02/11/00	Revision 2.5: 12/02/04
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(formerly CORP-IP0002NC)		Revision 2.2: 09/25/01		Revision 2.6: 07/29/07
		Revision 2.3: 01/18/02		Revision 0: 07/18/08 (NC-IP-010)
		Revision 2.4: 02/19/03		

16.2. Associated SOPs and Policies, latest version

16.2.1. Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Methods 6010B and 200.7, NC-MT-012

16.2.2. Inductively Coupled Plasma-Mass Spectrometry, EPA Methods 6020 and 200.8, NC-MT-002

16.2.3. TestAmerica North Canton Quality Control Program, QA-003

16.2.4. Glassware Washing, NC-QA-014

16.2.5. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.6. Method Detection Limits and Instrument Detection Limits, CA-Q-S-006 and NC-QA-021

16.2.7. Supplemental Practices for DOD Project Work, NC-QA-016

16.2.8. Standards and Reagents, NC-QA-017

17. **MISCELLANEOUS (TABLES, APPENDICES, ETC.)**

17.1. Method Deviations

Not applicable

APPENDIX A

TABLES

TABLE I

Method 3050A Approved Analyte List

Element	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

TABLE II
ICP Soil Matrix Spike and LCS Levels

Element	Working LCS/MS Standard (mg/L)	Soil MS/LCS Level * (mg/kg)
Aluminum	100	200
Antimony	25	50
Arsenic	100	200
Barium	100	200
Beryllium	2.5	5
Cadmium	2.5	5
Calcium	2500	5000
Chromium	10	20
Cobalt	25	50
Copper	12.5	25
Iron	50	100
Lead	25	50
Magnesium	2500	5000
Manganese	25	50
Molybdenum	50	100
Nickel	25	50
Potassium	2500	5000
Selenium	100	200
Silver	2.5	5
Sodium	2500	5000
Thallium	100	200
Vanadium	25	50
Zinc	25	50
Boron	50	100
Tin	100	200
Titanium	50	100

* Final soil spike concentration based on the addition of 2.0 mL working spike (Section 7.3) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE III
ICPMS Soil Matrix Spike and LCS Levels

Element	Working LCS/MS Standard (mg/kg)	Soil MS/LCS Level * (mg/kg)
Aluminum	1000	1000
Antimony	10	10
Arsenic	10	10
Barium	10	10
Beryllium	10	10
Cadmium	10	10
Calcium	1000	1000
Chromium	10	10
Cobalt	10	10
Copper	10	10
Iron	1000	1000
Lead	10	10
Magnesium	1000	1000
Manganese	10	10
Molybdenum	10	10
Nickel	10	10
Potassium	1000	1000
Selenium	10	10
Silver	10	10
Sodium	1000	1000
Strontium	10	10
Thallium	10	10
Vanadium	10	10
Zinc	10	10
Boron	10	10
Tin	10	10
Titanium	10	10

* Final soil spike concentration based on the addition of 1.0 mL working spike (Section 7.4) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE IV
Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples	Refer to determinative SOPs: NC-MT-002 NC-MT-012	Redigest and re-analyze samples
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples	Refer to determinative SOPs: NC-MT-002 NC-MT-012	Redigest and re-analyze all samples associated with the LCS
Matrix Spike	One per sample preparation batch of up to 20 samples	Refer to determinative SOPs: NC-MT-002 NC-MT-012	Reprep not required unless preparation error suspected
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: NC-MT-002 NC-MT-012	See Corrective Action for Matrix Spike

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept scrupulously clean.

Powdered gloves must not be used in the Metals Lab since the powder contains zinc, as well as other metallic analytes.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.


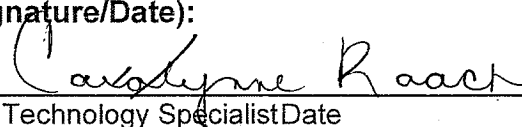
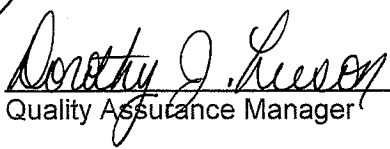
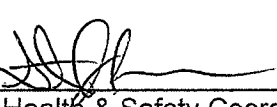


The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

**Title: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHODS
8000B, 8021B, 8081A, 8082, 8151A, 615 and 8015B**

Approvals (Signature/Date):			
	1/13/09		1-15-09
Technology Specialist	Date	Technology Specialist	Date
	1/12/09		1/12/09
Quality Assurance Manager	Date	Health & Safety Coordinator	Date
	1/12/09		1/16/09
Technical Director	Date	Laboratory Director	Date

This SOP was previously identified as SOP NC-GC-038, Rev 0, Dated 01/21/08

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1. SCOPE AND APPLICATION

- 1.1. This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices. See the list of appendices noted in the Table of Contents to determine the appropriate section of the SOP. Reporting limits are listed in each appendix.

2. SUMMARY OF METHOD

- 2.1. In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction or solid phase extraction (SOP NC-OP-032). Solid samples are prepared using sonication, Soxhlet or automated Soxhlet (PCB only) (SOP NC-OP-032). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).
- 2.2. After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

- 3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

- 4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See Appendices A through E for interferences specific to individual tests and suggested corrective actions. All glassware is cleaned per SOP NC-QA-014.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document.
- 5.2. Eye protection that prevents splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Refer to the TestAmerica North Canton Corporate Environmental Health and Safety Manual for a complete description of personal protection equipment. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately. Latex, Nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.
- 5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m ³ -TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
n-Propanol	Flammable Irritant	200 ppm-TWA	Causes irritation to the eyes, skin, and respiratory tract. May be absorbed through the skin. High concentrations can result in central nervous system depression. Severe exposure may cause respiratory depression, unconsciousness, convulsions, and death.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

- 5.4. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.
- 5.5. Exposure to chemicals must be maintained as low as reasonably achievable. All samples with stickers that read "Caution/Use Hood!" must be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.
- 5.6. Opened containers of neat standards must be handled in a fume hood.
- 5.7. Sample extracts and standards, which are in a flammable solvent, must be stored in an explosion-proof refrigerator.
- 5.8. When using hydrogen gas as a carrier, all precautions listed in the CSM must be observed.

- 5.9. Standard preparation and dilution must be performed inside an operating fume hood.
- 5.10. The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.11. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- 5.12. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

- 6.1. An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

- 7.1.1. Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for Method 8021B are stored at -10 to -20°C. Other stock standard solutions are stored as recommended by the manufacturer. All stock standards must be protected from light. Stock standard solutions must be brought to room temperature before using.
- 7.1.2. Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at least every week, unless the acceptability of the standard is demonstrated (Less than 20% drift from the initial calibration is an acceptable demonstration). Other volatile stock standards must be replaced every six months or sooner if comparison with check standards prepared from an independent source indicates a problem.
- 7.1.3. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If vendor-supplied standard has an earlier expiration date then that date is used. Refer to SOP NC-QA-017, Standards and Reagents, for additional information. The standard preparation information is detailed in the Standard Logbook.

7.2. Calibration Standards

7.2.1. Volatile Calibration Standards

- 7.2.1.1. The procedure for preparation of volatile standards is given in Appendix A.

7.2.2. Semivolatile Calibration Standards

- 7.2.2.1. Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at $\leq 6^{\circ}\text{C}$ and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

- 7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Zero Air.

7.4. Quality control (QC) Standards

- 7.4.1. QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

- 8.1. The holding time for semivolatile extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq 6^{\circ}\text{C}$. Volatile sample storage conditions and holding times are given in Appendix A.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

- 9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.
- 9.1.2. For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

- 9.2.1. Batches are defined at the sample preparation stage. Batches must be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica North Canton QC Program document (Policy QA-003) for further details of the batch definition. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC.
- 9.2.2. Quality Control Batch
- 9.2.2.1. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a LCSD may be substituted.

9.3. Control Limits

- 9.3.1. In-house historical control limits may be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined periodically. The recovery limits are mean recovery ± 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Project or program specific control limits may be used in place of in-house limits. Refer to Policy QA-003 for more details.
- 9.3.2. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries must be reported unless the dilution is more than 5X.

- 9.3.3. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes must be reported for all dilutions.
- 9.3.4. Refer to the QC Program document (Policy QA-003) for further details of control limits.
- 9.4. Surrogates
- 9.4.1. All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. Surrogate recoveries must be met in the method blank (MB) and Laboratory Check Samples (LCS or LCS/LCSD). If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):
- Check all calculations for error.
 - Ensure instrument performance is acceptable.
 - Recalculate the data and/or re-analyze the extract if either of the above checks reveals a problem.
 - The decision to re-analyze or flag the data must be made in consultation with the client. It is only necessary to reprepare / re-analyze a sample once to demonstrate poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.
- Note:** For DoD QSM, all surrogates must meet criteria. For Ohio VAP Projects, all surrogates must meet criteria unless the samples are ND and the surrogates are out high. Reanalysis or reparation of the samples is required if these criteria are not met.
- 9.4.2. If dual column analysis is used, the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in-control result is reported.
- 9.4.3. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and reparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reparation or flagging of the data is required. Reparation includes the parent sample and MS/MSD.
- 9.4.4. Refer to TestAmerica North Canton QC Program document (Policy QA-003) for further details of the corrective actions.
- 9.5. Method Blanks
- 9.5.1. For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatile samples and sodium sulfate for semivolatile soils tests (Refer to SOP NC-OP-032 for details). For low-level volatile soils, the method blank consists of reagent water and Ottawa sand. For medium-level volatile solids, the method blank consists of methanol and Ottawa sand. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client must take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

- 9.5.2. The method blank must not contain any analyte of interest at, or above, the reporting limit (except common laboratory contaminants, see below) or at, or above, 5% of the measured concentration of that analyte in the associated samples, whichever is higher.
- 9.5.3. If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.
- 9.5.4. Re-extraction and re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.
- 9.5.5. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be taken in consultation with the client.

Note: For Ohio VAP projects, the result must be below the reporting limit or samples must be re-extracted unless the samples are non-detect.
- 9.5.6. Refer to TestAmerica North Canton QC Program document (Policy QA-003) for further details of the corrective actions.
- 9.5.7. Refer to SOP NC-QA-016 for further details concerning DoD Project Work.
- 9.6. Laboratory Control Samples (LCS)
 - 9.6.1. For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes with a subset of control analytes. If any control analyte is outside the laboratory established historical control limits, corrective action must occur. All non-controlling compounds must attain a recovery of 5% or greater if the compound is on the client's list. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.
 - 9.6.2. If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project record and the report. (an example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable and the sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS.)
 - 9.6.3. If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.
 - 9.6.4. The LCS must have acceptable surrogate recoveries. If surrogate recoveries are low, re-extraction of the LCS and affected samples will normally be required. Consultation with the client should take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.
 - 9.6.5. Refer to TestAmerica North Canton QC Program document (Policy QA-003) for further details of the corrective action.
 - 9.6.6. If dual column analysis is used, the choice of which result to report is made in the same way as for samples (Section 12.1.2), unless one column is out of control, in which case the in control result is reported.
 - 9.6.7. LCS compound lists are included in the appendices.

9.7. Matrix Spikes

- 9.7.1. For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory-specific historically generated limits.
- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action must be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
 - If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action must normally include re-preparation and re-analysis of the batch.
 - If an MS/MSD is not possible due to limited sample, then an LCS duplicate must be analyzed. The recovery for each spike of the pair must be within established control limits. If the RPD is out of control, but both accuracy recoveries are within acceptance criteria, prepare an NCM and qualify report.
 - The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.
- 9.7.2. If dual column analysis is used, the choice of which result to report is made in the same way as for samples (Section 12.1.2), unless one column is out of control, in which case the in control result is reported.

9.8. Control Limits

- 9.8.1. Control limits are established by the laboratory as described in SOP, NC-QA-0018.
- 9.8.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs (QC Browser program).

10. CALIBRATION AND STANDARDIZATION

- 10.1. Internal or external calibration may be used. Internal calibration is recommended unless the sample matrix is likely to interfere with the quantitation of the internal standard. In either event, prepare standards containing each analyte of interest at a minimum of five concentration levels. The low-level standard must be at, or below, the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.
- 10.2. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.
- 10.3. With the exception of Section 10.4 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve, AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least five points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.
- 10.4. A level may be removed from the calibration if the reason can be clearly documented (for example, a broken vial or no purge run). A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be re-analyzed and the re-analysis used for the calibration. All initial calibration

points in a single calibration curve must be analyzed without any changes to instrument conditions, and all points in a single calibration curve must be analyzed within 24 hours.

10.5. External Standard Calibration

- 10.5.1. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$\text{Calibration Factor or Response Factor (CF) or (RF)} = \frac{\text{Area or Height of Peak}}{\text{Mass Injected (ng)}}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.6. Internal Standard Calibration

- 10.6.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If matrix interference is observed, reanalysis of the sample is required. If matrix interference is confirmed, report the second analysis and note in the Case Narrative. . For Ohio VAP projects, the laboratory must re-analyze any sample where the internal standard fails, and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution. If the internal standard is within criteria, report the second analysis. If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution. If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.

- 10.6.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows.

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured

A_{is} = Response for the internal standard

C_{is} = Concentration of internal standard

C_s = Concentration of the analyte to be determined in the standard

10.7. Calibration Curve Fits

- 10.7.1. Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is $\leq 20\%$. The average % RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. NOTE: This is not allowed for Ohio VAP projects.
- 10.7.2. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits must only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector. Linear or quadratic curve fits may be used if the compounds have historically exhibited a non-linear response.
- 10.7.3. Average Response Factor
- The average response factor may be used if the average percent relative standard deviation (% RSD) of all the response factors taken together is $\leq 20\%$.
- The equation for average response factor is:

$$\text{Average response factor} = \overline{RF} = \frac{\sum_{i=1}^n RF_i}{n}$$

Where: n = Number of calibration levels

$$\sum_{i=1}^n RF_i = \text{Sum of response factors for each calibration level}$$

- 10.7.4. Linear Regression
- The linear fit uses the following functions:
- 10.7.4.1. External Standard

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
 x = Concentration
 a = Slope
 b = Intercept

- 10.7.4.2. Internal Standard

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b \right]}{a}$$

Where: C_s = Concentration in the sample

A_s = Area of target peak in the sample

A_{is} = Area of internal standard in the sample

C_{is} = Concentration of the internal standard

10.7.5. Quadratic Curve

The quadratic curve uses the following functions:

10.7.5.1. External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.7.5.2. Internal Standard

$$y = a \left(\frac{A_s \times C_{is}}{A_{is}} \right) + c \left(\frac{A_s \times C_{is}}{A_{is}} \right)^2 + b$$

10.8. Evaluation of Calibration Curves

10.8.1. The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.8.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows.

$$\% RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^N \left[\frac{C_i - PC_i}{C_i} \right]^2}{(N - P)}}$$

Where:

N = Number of points in the curve

P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)

C_i = True concentration for level i

PC_i = Predicted concentration for level i

Note: When average response factors are used, % RSE is equivalent to % RSD.

10.9. The following requirements must be met for any calibration to be used.

- Response must increase with increasing concentration.
- If a curve is used, the calculated intercept of the curve at zero response must be less than \pm the reporting limit for the analyte.
- The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be $\leq 20\%$.
- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r . As a result, a curve may have a very good correlation coefficient (>0.995) while also having $> 100\%$ error at the low point.

10.10. Weighting of Data Points

10.10.1. In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and must be used if the data system has this capability.

10.11. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five-point initial calibration must be generated, and the sample(s) re-analyzed for quantitation.

10.12. Calibration Verification

10.12.1. 12-hour Calibration

10.12.1.1. The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence.

10.12.2. Calibration Verification

10.12.2.1. It may be appropriate to analyze a mid-point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12-hour calibration with the exception that retention times are not updated.

10.12.2.2. Any individual compounds with % D $< 15\%$ meet the calibration criteria. The calibration verification is also acceptable if the average of the % D for all the analytes is $< 15\%$. This average is calculated by summing the entire absolute % D results in the calibration (including surrogates) and dividing by the number of analytes. Only ND or results below the RL are reported. Any analyte that is reportable as found must have a % difference of $< 15\%$ in the calibration verification or 12-hour calibration on the column used for quantitation. Refer to Section 12.1.2 for which result to report.

- 10.12.3. An ICV is analyzed immediately after an initial calibration. The acceptance criteria is $\pm 15\%$ for TCL analytes and $\pm 30\%$ for all other analytes. If this is not met, a new initial calibration curve is analyzed.
- 10.12.4. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.
- 10.12.5. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. The bracketing standards on the column used for calibration must meet the same criteria as the opening standards. Bracketing is not necessary for internal standard methods.
- 10.12.6. If the analyst notes that a CCV has failed and can document the reason for failure (e.g., no purge, broken vial, carryover from the previous sample etc.), then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria, then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV, then the proceeding samples have not been successfully bracketed; but analysis may continue.
- 10.12.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed, then the first must serve as the bracketing standard for the preceding samples and the last must serve as the CCV for the following samples.
- 10.12.8. If highly contaminated samples are expected, it is acceptable to analyze blanks or primers at any point in the run.
- 10.12.9. Percent Difference Calculation

10.12.9.1. Percent difference for internal and external methods is calculated as follows:

Internal Standard

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

External standard

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where: RF_c and CF_c are the response and calibration factors from the continuing calibration

\overline{RF} and \overline{CF} are the average response & calibration factors from the initial calibration

10.12.10. Percent Drift Calculation

10.12.10.1. Percent drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for percent drift are the same as for percent difference

$$\% \text{ Drift} = \frac{\text{Calculated Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \times 100\%$$

10.12.10.2. Corrective Actions for Continuing Calibration

10.12.10.3. If the overall average percent drift of all analytes is greater than $\pm 15\%$, corrective action must be taken. This may include clipping the column, changing the liner, or other minor instrument adjustments, followed by re-analyzing the standard. If the overall average percent drift still varies by more than $\pm 15\%$, a new calibration curve must be prepared.

10.12.10.4. Corrective Action for Samples

10.12.10.4.1. For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be re-injected.

10.12.10.4.2. For external standard methods, any samples injected after the last good continuing calibration standard must be re-injected.

10.12.10.4.3. If the average percent drift for all the analytes in the calibration is over 15% ; but all of the analytes requested for a particular sample have percent drift $\leq 15\%$, then the analysis is acceptable for that sample.

11. PROCEDURE

11.1. Extraction

11.1.1. Extraction procedures are referenced in the SOP NC-OP-032, current revision.

11.2. Cleanup

11.2.1. Cleanup procedures are referenced in the SOP NC-OP-025, current revision.

11.3. Gas Chromatography

11.3.1. Chromatographic conditions for individual methods are presented in the appendices.

11.4. Sample Introduction

11.4.1. In general, volatiles analytes are introduced using Purge and Trap as described in Appendix A.

11.4.2. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

11.5.1. An analytical sequence starts with an initial calibration or a calibration verification. Refer to the individual method appendices (Appendices A, B, C, D, and E) for method-specific details of calibration verifications and analytical sequences.

11.5.2. The calibration verification includes analysis of standards containing all single response analytes and updating the retention time windows.

11.5.3. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a calibration verification.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). For multi-response analytes

(e.g., Aroclors), use the retention time of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

- 11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid-point of the initial calibration and each 12-hour calibration. The widths of the windows must remain the same until new windows are generated following the installation of a new column.
- 11.6.3. If the retention time window as calculated above is less than ± 0.05 minutes, use a retention time window appropriate for the analysis and run time. This allows for slight variations in retention times caused by sample matrix.
- 11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.
- 11.6.5. Retention time studies are filed in the laboratory.
- 11.6.6. Corrective Action for Retention Times
 - 11.6.6.1. The retention times of all compounds in the 12-hour calibration or calibration verification standard must be within the retention time window. If this condition is not met, all samples analyzed after the last compliant standard must be re-analyzed, unless the following conditions are met for any compound that elutes outside the retention time window.
 - 11.6.6.2. The retention time of that compound in the standard must be within a retention time range equal to twice the original window.
 - 11.6.6.3. No peak that would be reportable must be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.7. Daily Retention Time Windows

- 11.7.1. The center of the retention time windows determined in Section 11.6 is adjusted to the retention time of each analyte as determined in the 12-hour calibration standards or continuing calibration verification standards. (See Methods 8081A and 8082 Appendices B and C for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration or continuing calibration verification.

11.8. Procedural Variations

- 11.8.1. Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC Manager. The Nonconformance Memo must be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Procedural deviations are not allowed for Ohio VAP Projects.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column; but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases, GC/MS confirmation may be required. Client-specific requirements may also define the need for second column confirmation and/or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column at a concentration greater than the reporting limit (MDL if J flag confirmation required). Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. Many programs require chromatograms before and after manual integration. Additional information on manual integration can be found in SOP CA-Q-S-002.

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst:

- A) the primary column approach, or
- B) the better result approach

Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach

12.1.2.2. The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is > 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for re-analysis.)

12.1.2.3. Better result approach

The higher of the two results is normally reported. The higher result is considered better because the higher result is generally higher because of chromatographic interference. For Ohio VAP projects, both columns must meet calibration criteria. The lower result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the higher result
- The continuing or bracketing calibration on the column with the higher result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for re-analysis.)

12.1.3. If the Relative Percent Difference (RPD) between the response on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)}$$

Where R=Result

12.1.4. Multi-response Analytes

12.1.4.1. For multi-response analytes, the analyst must use the retention time window, but must rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst must weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

12.2.1. If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and re-analyzed. Dilutions must target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

12.3.1. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are detected, then the sample must be re-analyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

12.3.2.1. The most concentrated dilution with no target compounds above the calibration range must be reported. Other dilutions may be reported at client request if the lower dilutions will not cause detector saturation, column overload, or carryover. Analyst judgement and client site history will be factors in the reporting of dual dilutions.

12.4. Interferences

12.4.1. If peak detection is prevented by interferences, further cleanup must be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Internal Standard Criteria

12.5.1. If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration.

12.5.2. The internal standard response in samples must be within 50-150% of the response of previous continuing calibration standard.

12.6. Calculations

12.6.1. Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.6.2. External Standard Calculations

12.6.2.1. Aqueous samples

$$\text{Concentration (mg / L)} = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample

V_i = Volume of extract injected, μL

D_f = Dilution factor

V_t = Volume of total extract, μL

V_s = Volume of sample extracted or purged, mL

CF = Calibration factor, area or height/ng

12.6.2.2. Non-aqueous Samples

$$\text{Concentration (mg / kg)} = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W)}$$

Where:

W = Weight of sample extracted or purged, g

12.6.2.3. Internal Standard Calculations

12.6.2.4. Aqueous Samples

$$\text{Concentration (mg / L)} = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng

A_{is} = Response of the internal standard

RF = Response factor for analyte

12.6.2.5. Non-aqueous Samples

$$\text{Concentration (mg / kg)} = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

12.6.3. Surrogate Recovery

12.6.3.1. Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation.

$$\% \text{ Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

- 12.6.4. Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002

13. METHOD PERFORMANCE

13.1. Method Detection Limit

- 13.1.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOPs NC-QA-021 and CA-Q-S-006.

13.2. Initial Demonstration

- 13.2.1. Each laboratory must make a one-time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests, it may be necessary to use more than one QC check mix to cover all analytes of interest.
- 13.2.1.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample must be equivalent to a mid-level calibration.
- 13.2.1.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.
- 13.2.1.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

- 13.3.1. The Group/Team Leader has the responsibility to ensure an analyst who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

- 14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

- 15.1. All waste must be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate

Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention.”

15.2. Waste Streams Produced by the Method.

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Vials containing sample extracts. These vials are placed in the vial waste located in the GC/MS laboratory.

15.2.1.2. **Tubes containing sample extracts for TPH, Pesticides, PCBs and Herbicides.** These capped tubes are placed in the PCB/flammable waste located the GC prep laboratory.

15.2.1.3. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCB's must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB and PCB vial waste. PCB containing samples are located through a LIMS query and disposed of as PCB containing.

15.2.1.4. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane.** These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.1.5. **Discarded samples.** These samples are collected in the solid debris drum.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and Section 8000B

16.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.4. Revision History

Historical File:	Revision 2.1: 08/12/96	Revision 5.7: 10/01/03
(formerly CORP-GC-0001NC)	Revision 3.0: 12/01/97	Revision 5.8: 02/06/06
	Revision 5.3: 11/18/99	Revision 0: 01/21/08 (NC-GC-038)
	Revision 5.4: 11/10/00	
	Revision 5.5: 03/16/01	
	Revision 5.6: 05/25/01	

16.5. Associated SOPs and Policies, current version

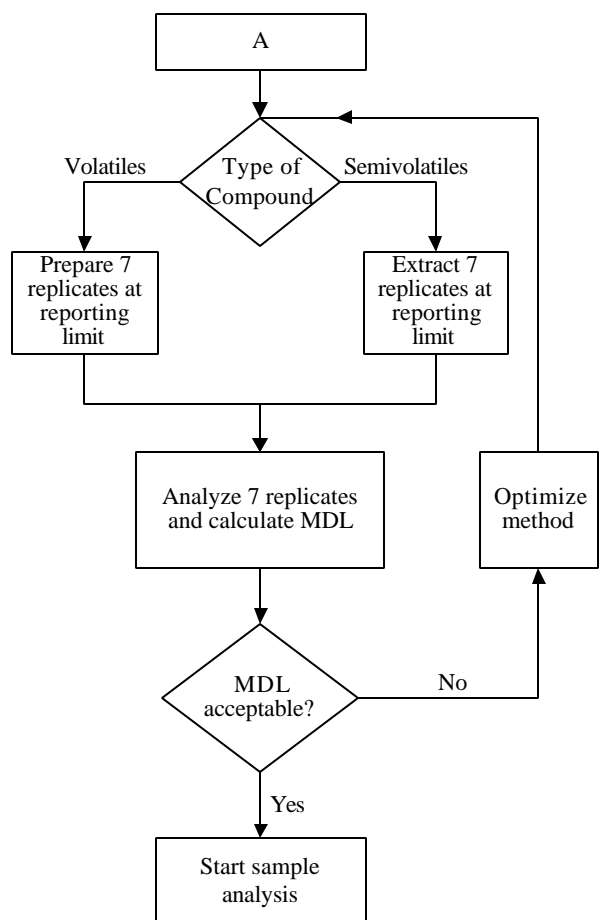
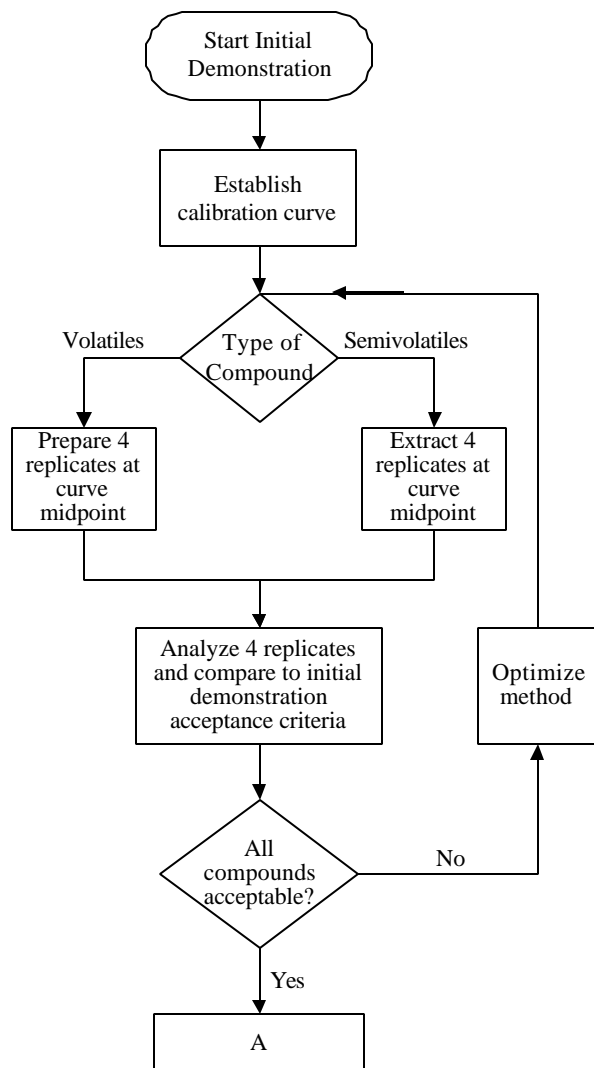
16.5.1 QA Policy QA-003

16.5.2 Glassware Washing, NC-QA-014

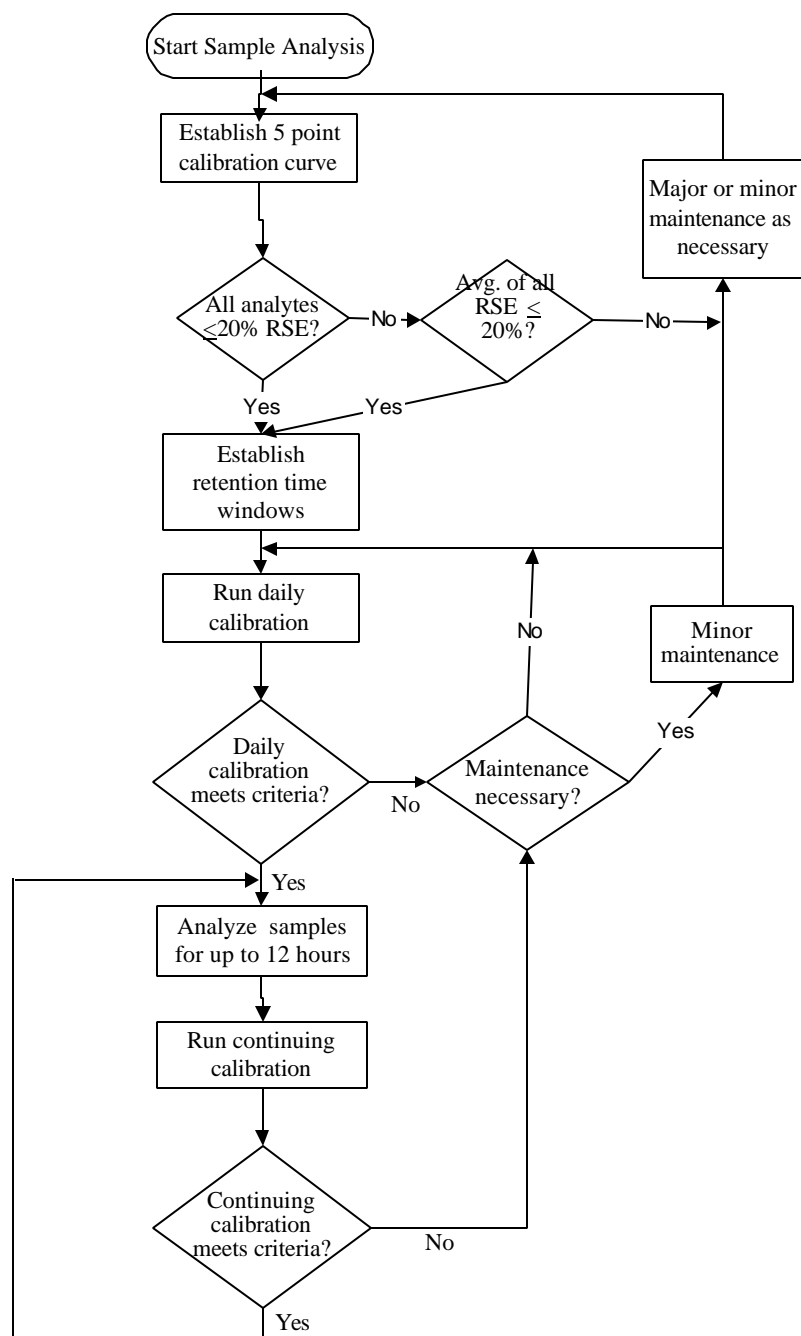
- 16.5.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018
- 16.5.4 Method Detection Limits and Instrument Detection Limits, CA-Q-S-006 and NC-QA-021
- 16.5.5 Standards and Reagents, NC-QA-017
- 16.5.6 Supplemental Practices for DoD Project Work, NC-QA-016
- 16.5.7 Acceptable Manual Integration Practices, CA-Q-S-002
- 16.5.8 Calibration Curves (General), CA-Q-S-005
- 16.5.9 Section of Calibration Points, CA-T-P-002

17. MISCELLANEOUS

- 17.1. Modifications from Reference Method
 - 17.1.1. Chapter 1 of SW-846 states the method blank must not contain any analyte of interest at, or above, the Method Detection Limit. This SOP states the Method Blank must not contain any analyte of interest at, or above, the reporting limit. Common lab contaminants are allowed to be up to five times the reporting limit in the blank following consultation with the client.
- 17.2. Flow Diagrams
 - 17.2.1. Initial demonstration and MDL



17.2.2 Sample Analysis¹



¹ This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

- 1.1. This SOP Appendix describes sample preparation and extraction for the analysis of volatile organics by a Purge and Trap procedure following Method 8021B. All requirements of the 8000B section of this SOP must be met except when superseded by this Appendix. Refer to Table A1 for the individual analytes normally determined by these procedures. Routine reporting limits are listed in Table A1.
- 1.2. Compounds within the scope of this method have boiling points below 200°C and are insoluble or slightly soluble in water. Classes of compounds best suited to Purge and Trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.
- 1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.
- 1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the Purge and Trap procedure.
- 1.5. The associated LIMS method code is QR.

2. SUMMARY OF METHOD

- 2.1. An inert gas is bubbled through the sample at ambient temperature or at 40°C (40°C required for low-level soils), and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and back-flushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a photo-ionization detector, an electrolytic conductivity detector, or a combination of both.
- 2.2. For soil samples, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by Purge and Trap GC following the normal water method.

3. DEFINITIONS

- 3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

- 4.1. Refer to Section 4 of the Method 8000B part of this SOP for general information on chromatographic interferences.
- 4.2. Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device must be avoided.
- 4.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

- 4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it must be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.
- 4.5. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every seven days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the seven-day period prior to the analysis of the contaminated holding blank is required. Refer to SOP NC-QA-020 for additional information on holding blanks.
- 4.6. Acidification of samples may result in hydrolysis of 2-chloroethyl-vinyl ether.

5. SAFETY

- 5.1. Refer to Section 5 of the Method 8000B section of this SOP for general safety requirements.
- 5.2. The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: Benzene, Carbon Tetrachloride, 1,4-Dichlorobenzene, 1,2-Dichloroethane, Hexachlorobutadiene, 1,1,2,2-Tetrachloroethane, 1,1,2-Trichloroethane, Chloroform, 1,2-Dibromoethane, Tetrachloroethene, Trichloroethene, Vinyl Chloride. Pure standard materials and stock standard solutions of these compounds must be handled in a hood.
- 5.3. GC VOA instruments use an ultraviolet (UV) light source, which must be shielded from view.

6. EQUIPMENT AND SUPPLIES

- 6.1. Microsyringes – 5 µL, 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL. These must be equipped with a 20 gauge (0.006" ID) needle. These must be used to measure and dispense methanolic solutions and aqueous samples.
- 6.2. Gas tight syringes -- 5 mL. Used for measuring sample volumes.
- 6.3. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.
- 6.4. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.
- 6.5. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A2 for suggested traps for specific tests.
- 6.6. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.
- 6.7. Columns -- Refer to Table A2 for details of columns
- 6.8. Volumetric flasks, Class A -- 5 mL to 250 mL
- 6.9. pH paper: Range 0-14
- 6.10. Balance capable of weighing to 0.01g for samples

- 6.11. Chlorine test strips
- 6.12. Hach chlorine test pillows
- 6.13. Mechanical pipettes: 10 mL and 20 mL

7. REAGENTS AND SUPPLIES

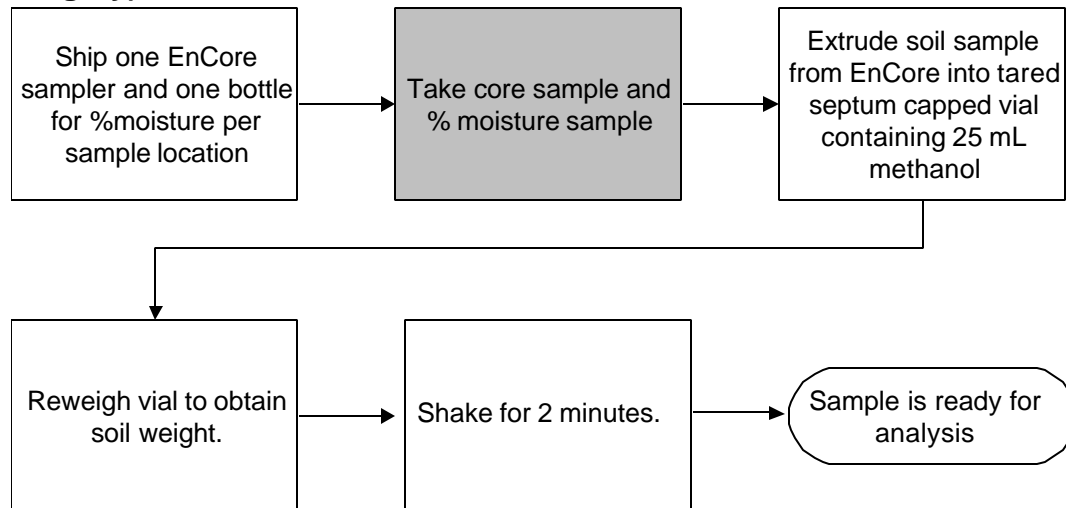
- 7.1. Refer to the Method 8000B section of this SOP for general requirements for reagents and supplies.
- 7.2. Organic-Free Water
 - 7.2.1. Organic-free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. The laboratory method for generating organic free water is continuously sparging water with helium or nitrogen.
- 7.3. Methanol -- Purge and Trap Grade
- 7.4. n-Propanol – ultra high
- 7.5. Standards
 - 7.5.1. Refer to Tables A3 for details of surrogate, matrix spiking, and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low-level standard must be equivalent to the reporting limits specified in Table A1.
 - 7.5.2. Volatile standards are prepared by injecting a measured volume of the stock standard into a volumetric flask containing the appropriate volume of methanol. See the Standards Logbook for details on sample preparation.

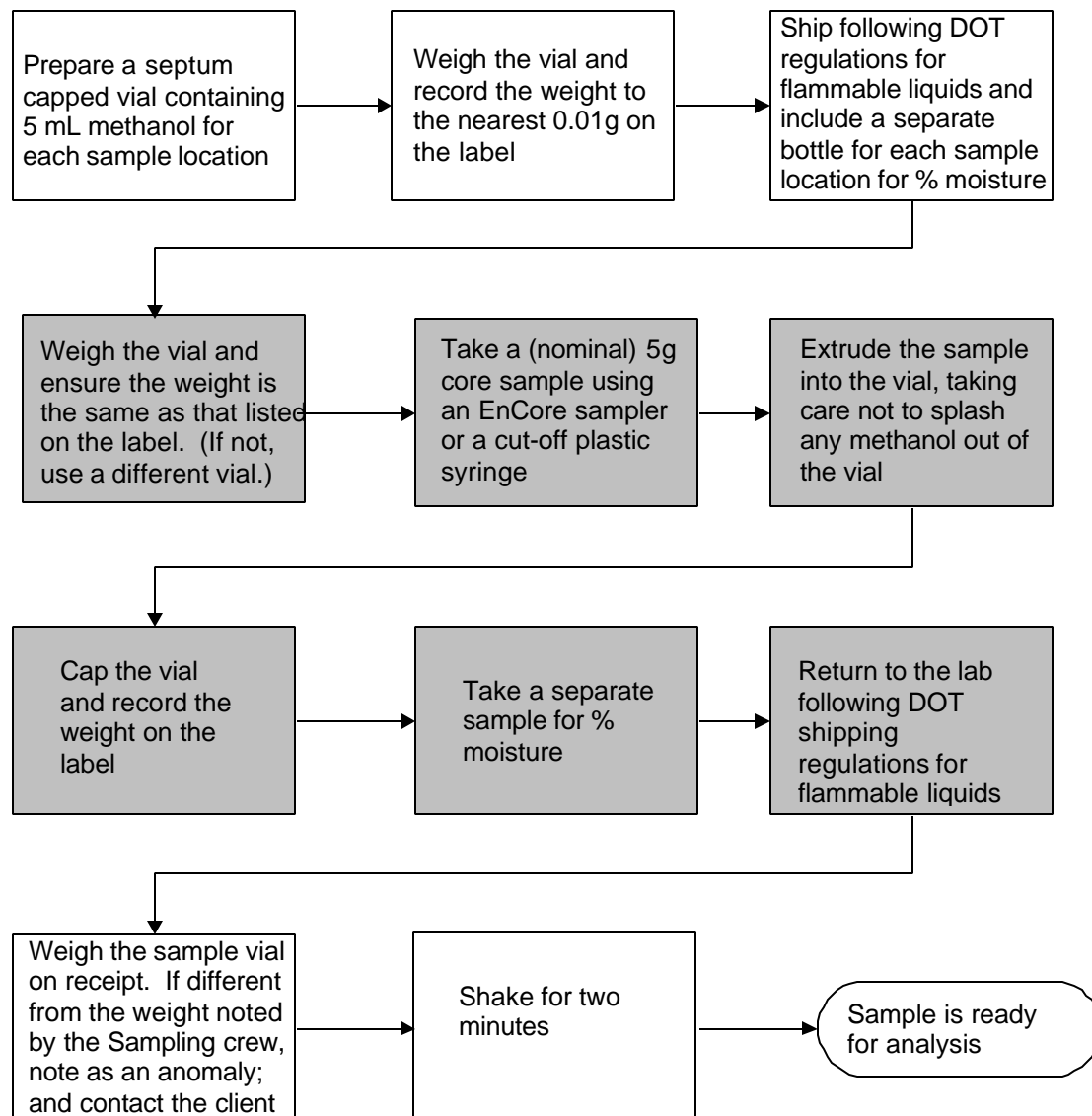
8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

- 8.1. Holding times for all volatile analysis are 14 days from sample collection to analysis.
- 8.2. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. Unpreserved samples must be analyzed within seven days from sample collection if aromatic compounds are requested.
- 8.3. Solid samples are field preserved with methanol for medium-level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.
- 8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5g or 25g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium-level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes), then it will be necessary to use two additional 5g EnCore™ samplers or to use field preservation.
- 8.5. Sample collection for medium-level analysis using EnCore™ samplers.
 - 8.5.1. Ship one 5g (or 25g) EnCore™ sampler per field sample position.
 - 8.5.2. An additional bottle must be shipped for percent moisture determination.
 - 8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25g sampler). Obtain the weight of

- the soil added to the vial and note on the label.
- 8.5.4. Add the correct amount of surrogate spiking mixture. (Add 1 µL of a 50 µg/mL solution for a nominal 5g sample, 500 µL for a medium level prep sample, 500 µL for a waste prep, and 2.5 mL for a 25g soil sample.)
 - 8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 10 µL of a 10 µg/mL solution for a nominal 5g sample, 500 µL for a medium level prep sample, 500 µL for a waste prep, and 2.5 mL for a 25g soil sample.) The addition of spike introduces a slight error (0.4%), which can be neglected, into the calculations.
 - 8.5.6. Prepare an LCS/LCSD for each batch by adding the correct amount of matrix spiking solution to clean methanol. (Add 10 µL of a 10 µg/mL solution for a nominal 5g sample, 500 µL for a medium level prep sample, 500 µL for a waste prep, and 2.5 mL for a 25g soil sample.)
 - 8.5.7. Method blanks are prepared using 5 g of Ottawa sand, 5mL of reagent waster, and a stir bar.
 - 8.5.8. Shake the samples for two minutes to distribute the methanol throughout the soil.
 - 8.5.9. Allow to settle and store in a clean Teflon®-capped vial at 4 +/-2°C until analysis.
 - 8.6. Sample collection for medium level analysis using field methanol preservation
 - 8.6.1. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.
 - 8.6.2. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25g sample, 20µL for a nominal 5g sample.)
 - 8.6.3. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25g sample, 20µL for a nominal 5g sample.) The addition of spike introduces a slight error (0.4%), which can be neglected, into the calculations.
 - 8.6.4. Prepare an LCS/LCSD for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).
 - 8.6.5. For medium level Method Blanks, prepare 5 mL of methanol, 50uL of surrogate and 5 g of Ottawa sand in a 20 mL vial.
 - 8.6.6. Shake the samples for two minutes to distribute the methanol throughout the soil.
 - 8.6.7. Allow to settle and store in a clean Teflon®-capped vial at 4 +/-2°C until analysis.
 - 8.7. Aqueous samples are stored in glass containers with Teflon®-lined septa at 4°C ± 2°C with minimum headspace. For volatile water samples, the method blank consists of reagent water
 - 8.8. Medium-level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon®-lined caps and stored at 4°C ± 2°C. The extracts are stored with minimum headspace.
 - 8.9. The maximum holding time is 14 days from sampling until the sample is analyzed. Maximum holding time for the EnCore™ sampler (before the sample is added to methanol) is 48 hours.
 - 8.10. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise, it is replaced every 14 days.

EnCore procedure when low level is not required (field steps in gray)



Field methanol extraction procedure (field steps in gray)

9. QUALITY CONTROL

- 9.1. Refer to the Method 8000B section of this SOP (Section 9) for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Refer to the Method 8000B section of this SOP (Section 10) for general calibration procedures.

10.2. Gas Chromatograph Operating Conditions

- 10.2.1. Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors. For simultaneous determination of aromatic and halogenated volatiles, a single column is used and the PID and ELCD detectors are connected in series.

- 10.2.2. Refer to Table A2, A3, and A4 for GC operating conditions. Additional operating instruction may be found in instrument manuals located in the laboratory.

10.3. Initial Calibration

- 10.3.1. Refer to Section 10 of the Method 8000B section of this SOP for details of initial calibration criteria.

- 10.3.2. Low-level soil samples must be purged at 40°C; therefore the calibration curve must also be purged at 40°C.

- 10.3.3. The low-level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector. See Table A5 for the I-cal level and amounts.

- 10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Initial Calibration Verification (ICV)

- 10.4.1. An initial calibration verification (ICV) standard is analyzed immediately following the initial calibration. Acceptance criteria is $\pm 15\%$ for TCL analytes and $\pm 30\%$ for all other analytes. If this is not met, a new initial calibration curve is analyzed.

10.5. Calibration Verification

- 10.5.1. A mid-level calibration standard is used for the calibration verification. The gases have 20% D criteria rather than the 15% used for other analytes. For analytes not listed in Method 8021B, the CCV criteria is 50% D.

- 10.5.2. A calibration verification run is performed after every ten samples or 12 hours for this method.

- 10.5.3. Bracketing of samples with calibration verification runs is only necessary for external standard analysis. Analytes detected using a photo-ionization detector are quantitated using the internal standard method. Analytes detected using an electrolytic conductivity detector may be quantitated using either the internal or external method.

11. PROCEDURE

- 11.1. Refer to the Method 8000B section of this SOP for general procedural requirements.

11.2. Analytical Sequence

- 11.2.1. The analytical sequence starts with an initial calibration of at least five points or a 12-hour calibration that meets % difference criteria from an existing initial calibration.

11.3. Confirmation

- 11.3.1. The PID and ELCD detectors are sufficiently selective that second column confirmation is not always necessary. Requirements for second column confirmation must be decided in consultation with the client. If the PID and ELCD are used in series, confirmatory information for many analytes can be gained by comparing the relative response from the two detectors.

11.4. Aqueous and Soil Sample Analysis (Autosampler Purge and Trap units that sample directly from the VOA vial)

- 11.4.1. Check the pH of the sample in the VOA vial prior to analysis. Samples are also checked for residual chlorine at this time.
- 11.4.2. Units, which sample from the VOA vial, should be equipped with a module, which automatically adds surrogate and internal standard solution, as needed, to the sample prior to purging the sample.
- 11.4.3. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial.
Note: Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.
- 11.4.4. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.5. Low-Level Solids Analysis using discrete autosamplers Bulk Solids

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

- 11.5.1. This procedure cannot be used for Ohio VAP samples. Refer to Section 1.4 for the appropriate procedure.
- 11.5.2. Do not discard any supernatant liquids. Mix the contents of the container
- 11.5.3. *Weigh out 5g (or other appropriate aliquot) of sample into a disposable culture tube or 40mL vial. Record the weight to the nearest 0.1g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5g. If the sample is contaminated with analytes such that a purge amount less than 0.5g is appropriate, use the medium-level method described in Section 8.6, Appendix A.*
- 11.5.4. *Place in autosampler.*
- 11.5.5. *Add 5 mL of organic free water to each vial. Add surrogate/internal standard (and matrix spike solutions if required.) (See Table A3.) Add directly to the sample from Section 11.6.2.*

11.5.6. *The above steps must be performed rapidly and without interruption to avoid loss of volatile organics*

11.6. Methanol Extract Soils

11.6.1. Take an appropriate aliquot from the sample prep container. Add to 5mL of organic free water in a 40 mL VOA vial. Turn off the automatic surrogate function on the autosampler. Place the sample in the autosampler and analyze as for aqueous samples. If less than 1µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1µL will be added to the water in the VOA vial.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to Section 12 of the Method 8000B section of this SOP. Refer to Tables A5 and A6 for details of the calibration curves.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the Method 8000B section of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the 8000B section of this SOP

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the 8000B section of this SOP

15.2. Waste streams produced by the method.

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. **Acidic material from the auto-sampler.** Waste stream must be collected and neutralized before discharge to a sewer system if the pH is less than 4.

15.2.1.2. **Methanol waste standards.** Methanol waste is discarded as a flammable liquid.

15.2.1.3. **All samples including purged and extracted soils and waters:** Samples are collected in boxes and removed from the lab to storage. The waste coordinator handles crushing the vials and proper disposal.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035 and 8021B.

16.2. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Rev 0, December 1996.

16.3. Laboratory Holding Blanks, NC-QA-020

17. MISCELLANEOUS

17.1. TABLES

Table A1				
Standard Analyte List for Method 8021B				
Compound	CAS No.	Reporting Limit, µg/L or µg/kg		
		Aqueous	Low Soil	Medium Soil
1,1,1,2-Tetrachloroethane	630-20-6	1.0		
1,1,1-Trichloroethane	71-55-6	1.0		
1,1,2,2-Tetrachloroethane	79-34-5	1.0		
1,1,2-Trichloroethane	79-00-5	1.0		
1,1-Dichloroethane	75-34-3	1.0		
1,1-Dichloroethene	75-45-4	1.0		
1,1-Dichloropropene	563-58-6	1.0		
1,2,3-Trichlorobenzene	87-61-6	1.0		
1,2,3-Trichloropropane	96-18-4	1.0		
1,2,4-Trichlorobenzene	120-82-1	1.0		
1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	50
1,2-Dibromo-3-Chloropropane(DBCP)	96-12-8	1.0		
1,2-Dibromoethane(EDB)	106-93-4	1.0		
1,2-Dichlorobenzene	95-50-1	1.0		
1,2-Dichloroethane	107-06-2	1.0		
1,2-Dichloropropane	78-87-5	1.0		
1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	50
1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
1,3-Dichloropropane	142-28-9	1.0		
1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
2,2-Dichloropropane	590-20-7	1.0		
2-Chloroethyl vinyl ether	110-75-8	5.0		
2-Chlorotoluene	95-49-8	1.0		
4-Chlorotoluene	106-43-4	1.0		
Acetone	67-64-1	10		
Benzene	71-43-2	1.0	1.0	50
Benzyl Chloride	100-44-7	5.0		
Bromobenzene	108-86-1	1.0		
Bromochloromethane	74-97-5	1.0		
Bromodichloromethane	75-27-4	1.0		
Bromoform	75-25-2	1.0		
Bromomethane	74-83-9	1.0		
Carbon Tetrachloride	56-23-5	1.0		
Chlorobenzene	108-90-7	1.0		
Chlorodibromomethane	124-48-1	1.0		
Chloroethane	70-00-3	1.0		

Table A1				
Standard Analyte List for Method 8021B				
Compound	CAS No.	Reporting Limit, µg/L or µg/kg		
		Aqueous	Low Soil	Medium Soil
Chloroform	67-66-3	1.0		
Chloromethane	74-87-3	1.0		
cis-1,2 Dichloroethene	156-59-4	1.0		
cis-1,3-Dichloropropene	10061-01-5	1.0		
Dibromomethane	74-95-3	1.0		
Dichlorodifluoromethane	75-71-8	1.0		
Ethyl Benzene	100-41-4	1.0	1.0	50
Freon 113	76-13-1	1.0		
Hexachlorobutadiene	87-68-3	1.0		
Isopropylbenzene	98-82-8	1.0		
MEK (2-butanone)	78-93-3	5.0		
Methyl tert-butyl ether (MTBE)	1634-04-4	1.0		
Methylene Chloride	75-09-2	5.0		
MIBK (4-methyl-2-pentanone)	108-10-1	5.0		
Naphthalene	91-20-3	2.0	2.0	250
n-butylbenzene	104-51-8	1.0		
n-Propylbenzene	10306501	1.0		
p-Isopropyltoluene	99-87-6	1.0		
sec-Butylbenzene	135-98-8	1.0		
Styrene	100-42-5	1.0		
tert-Butylbenzene	98-06-6	1.0		
Tetrachloroethene	127-18-4	1.0		
Toluene	108-88-3	1.0	1.0	50
trans-1,2-Dichloroethene	156-60-5	1.0		
trans-1,3-Dichloropropene	10061-02-6	1.0		
Trichloroethene	79-01-6	1.0		
Trichlorofluoromethane	75-69-4	1.0		
Vinyl Chloride	75-01-4	1.0		
Xylenes (total)	1330-20-7	1.0	1.0	50

Table A2 Recommended Conditions for Method Combined Aromatic and Halogenated Volatiles	
Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	RTX - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Hydrogen
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A3 Surrogate and Internal Standard Concentrations				
Standard	Components	Working Solution µg/mL	Spike amount µL	Final concentration µg/L (µg/kg)
Combined aromatic and halogenated volatiles IS/SS	Fluorobenzene (SS)	50	1	10
	1,4-Dichlorobutane (SS)	50	1	10
	a,a,a-Trifluorotoluene	50	1	10

It may be necessary to select different surrogates in order to minimize sample interferences. 1-chloro-4-fluorobenzene is fairly well resolved from analytes listed in this SOP. Other surrogates that may be considered, and issues associated with their use are:

1-Chloro-2-fluorobenzene:	Elutes close to ethylbenzene on DB-1 or Rtx-1 and close to m,p-xylene on 502.2
Bromofluorobenzene:	Close to 1,1,2,2-trichloroethane and 1,2,3-trichloropropane on the 502.2 column. Good on DB-1 or Rtx-1.
2-Bromo-1-chloropropane:	May coelute with 1,1,2-trichloroethane

Table A4				
Concentrations for LCS and MS/MSD compounds				
Standard	Components	Working Solution µg/mL	Spike amount µL	Final concentration µg/L (µg/kg)
Aromatic	Benzene	10	10	20
	Toluene	10		20
	Chlorobenzene	10		20
Halogenated	Chlorobenzene	10	10	20
	1,1-Dichloroethene	10		20
	Trichloroethene	10		20
Combination aromatic / Halogenated	Benzene	10	10	20
	Toluene	10		20
	Chlorobenzene	10		20
	1,1-Dichloroethene	10		20
	Trichloroethene	10		20

Table A5		
Initial Calibration for Water Analysis		
Cal Level	On Column Amount (ng)	Final Concentration (ng/L)
1	2.5	0.5
2	5	1
3	10	2
4	20	4
5	50	10
6	100	20
8	200	40
10	400	80

Table A6		
Soil or Water Heated Initial Calibration Curve		
Cal Level	On Column Amount (ng)	Final Concentration (ng/L)
1	2.5	0.5
2	5	0.1
3	10	2
5	50	10
6	100	20
7	200	40
8	400	80

1. SCOPE AND APPLICATION

- 1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8081A is applied to the analysis of organochlorine pesticides by GC/ECD. This appendix may also be applied when discontinued SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOPs (NC-OP-032).
- 1.2. Table B1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.
- 1.3. At client request, this method may also be used for the analysis of PCBs (Aroclors) in combination with pesticides, although these are normally analyzed following Method 8082, as described in Appendix C of this SOP. Extracts that have been acid cleaned may not be analyzed for pesticides, since several of the pesticides will be degraded.
- 1.4. The associated LIMS method code is QJ (Method 8081A).

2. SUMMARY OF METHOD

- 2.1. This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation is by external standard methods.

3. DEFINITIONS

- 3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

- 4.1. Refer to the Method 8000B section of this SOP for information regarding chromatographic interferences.
- 4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.
- 4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-025, Cleanup SOP.
- 4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Using hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

- 5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.
- 5.2. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and the BHCs. Primary standards of these toxic compounds must be prepared in a hood.
- 5.3. All ⁶³Ni sources must be leak-tested every six months, or in accordance with the manufacturer's general radioactive material license.

- 5.4. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the Director, EH&S must be immediately notified, and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the Method 8000B section of this SOP. A ^{63}Ni electron capture detector is required.
- 6.2. Refer to Table B2 for analytical columns.
- 6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.
- 6.4. Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

- 7.1. Refer to the Method 8000B section of this SOP for general requirements for reagents and supplies.
- 7.2. Refer to Table B3 for details of calibration standards. See the Standards Logbook for details on sample preparation.
- 7.3. Surrogate Standards
- 7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to Tables B5 and B6 for details of surrogate standards.
- 7.4. Column Degradation Evaluation Mix
- 7.4.1. A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. The holding time for pesticide extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq 6^{\circ}\text{C}$.

9. QUALITY CONTROL

- 9.1. Refer to the Method 8000B section of this SOP (Section 9) for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Refer to Section 10 of the Method 8000B section of this SOP for general calibration requirements.
- 10.2. Refer to Table B2 for recommended details of GC operating conditions. The conditions listed must result in resolution of all analytes listed in Table B1 on both columns.
- 10.3. Column Degradation Evaluation

- 10.3.1. Before any calibration runs, either initial or 12 hour, the column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see Equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.
- 10.3.2. If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:
- 10.4. Replacement of the injection port liner or the glass wool.
- 10.5. Cutting off a portion of the injection end of a capillary column.
- 10.6. Replacing the GC column.
- 10.7. Initial Calibration
 - 10.7.1. Refer to Section 10 of the Method 8000B section of this SOP for details of calibration procedures.
 - 10.7.2. Refer to Table B7 for the initial calibration analytical sequence.
 - 10.7.3. The response for each single-peak analyte must be calculated by the procedures described in the general method for GC analysis.
 - 10.7.4. The surrogate calibration curve is calculated from the AB mix. If there are resolution problems, then the A and B mixes may be analyzed separately.
 - 10.7.5. For multi-component pesticides:
 - 10.7.5.1. A five-point calibration is used for multi-component pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to Section 12.3 for guidance on which option to use.
 - 10.7.5.2. A full five-point calibration for any of the multi-component analytes is analyzed.
- 10.8. 12-hour Calibration Verification
 - 10.8.1. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration. A mid-level calibration standard is used for the 12-hour calibration. Refer to the Method 8000B section of this SOP for acceptance criteria.
 - 10.8.2. At a minimum, the 12-hour calibration includes analysis of the breakdown mix followed by mid-level standards of the AB mix. At a minimum, multi-component analytes are analyzed at the beginning of a sequence.
 - 10.8.3. The retention time windows for any analytes included in the 12-hour calibration are updated.
- 10.9. Initial Calibration Verification (ICV)

- 10.9.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. Acceptance criteria is $\pm 15\%$ for TCL analytes and $\pm 30\%$ for all other analytes. If this is not met, a new initial calibration curve is analyzed.

10.10. Continuing Calibration

- 10.10.1. A mid-level AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse, analyze the 12-hour standard sequence instead. The continuing calibration standard need not include multi-component analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

11. PROCEDURE

- 11.1. Refer to the Method 8000B section of this SOP for general procedural requirements.
- 11.2. Extraction
- 11.2.1. The extraction procedure is described in SOP NC-OP-032.
- 11.3. Cleanup
- 11.3.1. Cleanup procedures are described in SOP No. NC-OP-025.
- 11.4. Suggested gas chromatographic conditions are given in Table B2.
- 11.5. Allow extracts to warm to ambient temperature before injection.
- 11.6. The suggested analytical sequence is given in Table B7.

12. DATA ANALYSIS AND CALCULATIONS

- 12.1. Refer to the Method 8000B section of this SOP for identification and quantitation of single component analytes.
- 12.2. Identification of Multi-Component Analytes
- 12.2.1. Retention time windows are also used for identification of multi-component analytes, but the “fingerprint” produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.
- 12.3. Quantitation of Multi-Component Analytes
- 12.3.1. Use 3-10 major peaks (or total area for quantitation) as described in Section 10.4.4, initial calibration of multi-component analytes.
- 12.3.2. If there are no interfering peaks within the envelope of the multi-component analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.
- 12.3.2.1. Multiple peak option

- 12.3.3. This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e., peaks which are significantly larger than would be expected from the rest of the pattern.)
- 12.3.4. Chlordane may be quantitated either using the multiple peak option (Section 12.3.1, Appendix B), total area option (Section 12.3. 2., Appendix B), or by quantitation of the major components, α -chlordane, γ -chlordane and heptachlor.
- 12.4. Total area option
- 12.4.1. The total area of the standards and samples may be used for quantitation of multi-component analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option must not be used if there are significant interference peaks within the multi-component pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.
- 12.5. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence. For Ohio VAP projects, both columns must meet criteria.
- 12.6. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.
- 12.7. Calculation of Column Degradation/% Breakdown (%B)

Equation 9

$$12.7.1. \text{ DDT \%B} = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

Where:

A_{DDD} , A_{DDE} , and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

Equation 10

$$\text{Endrin \%B} = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_E} \times 100$$

Where:

A_{EK} , A_{EA} , and A_E = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

- 13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

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14.1. Refer to Section 14 of the Method 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the 8000B section of this SOP.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8081A

17. MISCELLANEOUS

17.1. Modifications from Reference Method - None

17.2. TABLES

Table B1 Standard Analyte List and Reporting Limits for Method 8081A			
Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
APPENDIX IX ADD-ONS			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzilate	0.1	3.3	100
<i>Kepone</i> ¹	1.0	33	1000

¹ Kepone is sometimes requested for analysis by method 8081A. However, kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result kepone analysis by 8081A is unreliable and not recommended. Analysis by method 8270C is a possible alternative.

Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects. The following concentration factors are assumed in calculating the Reporting Limits:

	<u>Extraction Vol.</u>	<u>Final Vol.</u>
Ground water	1000 mL	10 mL
Low-level Soil	30g	10 mL
High-level soil / waste	1g	10 mL

Table B2 Recommended Conditions for Method 8081A	
Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	Rtx-CLPesticides 30m x 0.32mm id, 0.5µm
Column 2	Rtx-35 30m x 0.32 mm id, 0.5µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B3 Calibration Levels ng/mL for Method 8081A						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6 ²
Individual Mix AB¹						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	5	10	25	50	100	200
δ-BHC	5	10	25	50	100	200
α-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane ³	5	10	25	50	100	200
γ-Chlordane ³	5	10	25	50	100	200
Multi-component Standards						
Chlordane (Technical)	20	50	100	200	500	
Toxaphene	200	500	1000 ⁵	2000	5000	
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

¹ Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.² Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.³ Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

Table B4	
Column Degradation Evaluation Mix ng/mL for Method 8081A	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B5			
LCS/Matrix Spike and Surrogate Spike levels mg/L or mg/kg for Method 8081A			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	33.3	200
Aldrin	0.20	33.3	200
Heptachlor	0.20	33.3	200
Dieldrin	0.50	33.3	500
Endrin	0.50	33.3	500
4,4'-DDT	0.50	33.3	500
Tetrachloro-m-xylene (Surrogate)	0.20	33.3	200
Decachlorobiphenyl (Surrogate)	0.20	33.3	200

Table B6		
LCS/Matrix Spike and Surrogate Spike levels for TCLP mg/L or mg/kg for Method 8081A		
	Aqueous	Waste
Heptachlor	5	500
Heptachlor epoxide	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table B7
Suggested Analytical Sequence for Method 8081A

Initial Calibration

ICV	
Solvent blank (optional)	
Primer if needed	
Breakdown Mix	
Individual mix AB	All levels
Technical Chlordane	Level 3 ¹
Toxaphene	Level 3 ¹
Up to 20 samples unless 12 hours comes first)	
Solvent blank (optional)	
Individual mix AB	Mid level (Continuing calibration)
Samples	
After 12 hours:	
Breakdown mix	
Individual mix AB	
Any other single component analytes	
Any multi-component analytes	

¹ A five-point curve for any of the multi-component analytes may be included. If Aroclors are included, a five-point calibration for Aroclor 1016/1260 must be included with the initial calibration and a single point for the other Aroclors. The mid point 1016/1260 mix is included with the daily calibration (every 12 hours).

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

Note: The initial primer is used if the instrument has been idle for a period of time.

12 -Hour Calibration

At least every 12 hours, counting from the start of the initial calibration or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB; and the breakdown mix must be run before the continuing calibration.

1. SCOPE AND APPLICATION

- 1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8082 is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This Appendix is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOP (NC-OP-032). PCBs are determined and quantitated as Aroclor mixes.
- 1.2. Tables C1 and C5 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

Note: SW-846 method 8082 provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

- 1.3. The associated LIMS method code is QH (8082).

2. SUMMARY OF METHOD

- 2.1. This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

- 3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

- 4.1. Refer to the Method 8000B section of this SOP for information regarding chromatographic interferences.
- 4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.
- 4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-025.
- 4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts. These cleanup procedures are included in SOP NC-OP-025.

5. SAFETY

- 5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.
- 5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.
- 5.3. All ⁶³Ni sources must be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

- 5.4. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the EH&S Director must be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the Method 8000B section of this SOP. A ^{63}Ni electron capture detector is required.
- 6.2. Refer to Table C2 for analytical columns.
- 6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

- 7.1. Refer to the Method 8000B section of this SOP for general requirements for reagents and supplies.
- 7.2. Refer to Table C3 for details of calibration standards. See the Standards Logbook for details on sample preparation.
- 7.3. Surrogate Standards
- 7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to Table C4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. The holding time for PCB extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq 6^{\circ}\text{C}$.

9. QUALITY CONTROL

- 9.1. Refer to the Method 8000B section of this SOP (Section 9) for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Refer to Section 10 of the Method 8000B section of this SOP for general calibration requirements.
- 10.2. Initial Calibration
- 10.2.1. Refer to Table C6 for the initial calibration analytical sequence.
- 10.2.2. The response for each Aroclor must be calculated by the procedures described in the general method for GC analysis with the following modifications.
- 10.2.3. A minimum five-point calibration of all Aroclors is generated. The average response factor is used to quantitate Aroclors. The low-level standard must be at or below the reporting limit. The other standards define the working range of the detector.
- 10.2.4. The high and low standards for the initial five-point calibration of 1016 / 1260 define the acceptable quantitation range for the other Aroclors. If any Aroclor is determined above this concentration, the extract must be diluted and re-analyzed.
- Note:** For Ohio VAP, Aroclor 1268 may be analyzed. In order to meet project-specific reporting limits, a lower concentration standard may be added to the calibration curve.
- 10.2.5. If the analyst knows that a specific Aroclor is of interest for a particular project, that Aroclor

may be used for the five point calibration rather than the 1016 / 1260 mix.

10.2.6. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.7. The following is used for the quantitation of all Aroclors. The same quantitation option must be used for standards and samples.

10.2.7.1. Multiple peak option.

10.2.7.2. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks.

10.3. 12-Hour Calibration

10.3.1. The 12-hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration.

10.3.2. At a minimum, the 12-hour calibration includes analysis of the Aroclor 1260 / 1016 mix.

10.3.3. Other Aroclors are included in the daily calibration check.

10.3.4. The retention time windows for any analytes included in the daily calibration and CCVs are updated.

10.3.5. For this method, samples must be bracketed with successful calibration verification runs.

10.4. Initial Calibration Verification (ICV)

10.4.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. Acceptance criteria is $\pm 15\%$. If this is not met, a new initial calibration curve is analyzed.

10.5. Calibration Verification Standards

10.5.1. The Aroclor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.)

10.5.2. A mid-level standard is used for the calibration verification.

11. PROCEDURE

11.1. Refer to the Method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

11.2.1. The extraction procedure is described in SOP NC-OP-032.

11.3. Cleanup

11.3.1. Cleanup procedures are described in SOP NC-OP-025.

11.4. Suggested gas chromatographic conditions are given in Table C2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table C6.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Identification of Aroclors

12.1.1. Retention time windows are used for identification of Aroclors, but the “fingerprint” produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.1.2. A clearly identifiable Aroclor pattern serves as confirmation of single column GC analysis. Dual column confirmation may be used for specific program requirements or by client request.

12.2. Quantitation of Aroclors

12.2.1. Use 3-10 major peaks or total area for quantitation

12.2.2. If the analyst believes that a combination of Aroclor 1254 and 1260 or a combination of 1242, 1248 and 1232 is present, then only the predominant Aroclor is quantitated and reported; but the suspicion of multiple Aroclors is discussed in the narrative. If well-separated Aroclor patterns are present, then multiple Aroclors may be quantitated and reported.

12.3. Second column confirmation of Aroclors will only be performed when requested by the client. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence. For Ohio VAP projects, both columns must meet criteria.

12.4. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits, or if one is <10%.

Note: For Ohio VAP samples and DoD projects, all surrogates must meet acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

13.2. Method detection limits (MDL) are determined for all Aroclors.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the Method 8000 section of this SOP

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8082

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. TABLES

Table C1 Standard Analyte list and Reporting Limits for Method 8082			
Compound	Reporting Limit, µg/L or µg/kg		
	Water	Soil	Waste
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	<u>Extraction Vol.</u>	<u>Final Vol.</u>
Ground water	1000 mL	10 mL
Low-level Soil	30g	10 mL
High-level soil / waste	1g	10 mL

Table C2 Instrumental Conditions for Method 8082	
Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm
Column 2	DB-1701 or Rtx 1701 30m x 0.32 mm id, 0.25µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table C3 Calibration Levels ng/mL for Method 8082						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6 ¹
Aroclor 1016/1260	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1242 ²	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1221 +1254 ²	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1232 ²	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1248 ²	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1262	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1268	0.05	0.1	0.2	0.5	1.0	2.0
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

¹ Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

² Aroclors may be quantitated within the range 100 to 2000 ng/mL (4000ng/mL if the level 6 1016/1260 standard is included). If the Aroclor is more concentrated, it must be re-analyzed at a dilution.

Table C4 LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis mg/L or mg/kg for Method 8082			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table C5 Michigan Analyte List and Reporting Limits¹ for Method 8082			
Compound	Reporting Limit		
	water (µg/L)	soil (µg/Kg)	
Aroclor-1016	0.2	330	
Aroclor-1221	0.2	330	
Aroclor-1232	0.4	330	
Aroclor 1242	0.2	330	
Aroclor-1248	0.2	330	
Aroclor-1254	0.2	330	
Aroclor-1260	0.2	330	

¹ Reporting Limits are only for samples performed under the Michigan program

Table C6
Suggested Analytical Sequence for Method 8082

Initial Calibration

Injection #

1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
8	Aroclor 1242	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
9	Aroclor 1248	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
10	Aroclor 1221/1254	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
11	Aroclor 1268 or 1262	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
12	ICV	
13-32	Sample 1-20 (or as many samples as can be analyzed in 12 hours)	
33	Aroclor 1016/1260	Level 3

etc

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

12-hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1260 / 1016 mix. Mid level standards of any other Aroclors expected to be present in the samples are also injected.

1. SCOPE AND APPLICATION

- 1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8151A or EPA Method 615 is applied to the analysis of chlorinated phenoxy acid herbicides in extracts prepared by SOP NC-OP-
- 1.2. The herbicides listed in Table D1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in Section 9 and the initial demonstration of method performance in Section 13 are met. Routine reporting limits are listed in Table D1.
- 1.3. The associated LIMS method codes are QS (8151A) and HR (615).

2. SUMMARY OF METHOD

- 2.1. This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

3. DEFINITIONS

- 3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

- 4.1. Refer to the Method 8000B section of this SOP for general information regarding chromatographic interferences.
- 4.2. Chlorinated acids and phenols cause the most direct interference with this method.
- 4.3. Sulfur may interfere and may be removed by the procedure described in SOP NC-OP-025.

5. SAFETY

- 5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.
- 5.2. All ⁶³Ni sources must be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.
- 5.3. All ⁶³Ni sources must be inventoried every six months. If a detector is missing, the EH&S Director must be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the Method 8000B section of this SOP. A Ni₆₃ electron capture detector is required.
- 6.2. Refer to Table D2 for analytical columns.
- 6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

- 7.1. Refer to Section 7 of the Method 8000B section of this SOP for general information on reagents and standards.

7.2. Refer to Table D4 for details of calibration standards. See the Standards Logbook for details on sample preparation.

7.3. Surrogate Standards

7.3.1. DCAA is the surrogate standard. Refer to Table D4 for details of surrogate standards.

8. SAMPLE PREPARATION, PRESERVATION, AND STORAGE

8.1. The holding time for herbicide extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq 6^{\circ}\text{C}$.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the Method 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table D3 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the Method 8000B section of this SOP for general calibration requirements.

10.2. Calibration standards are prepared from purchased standards in the methyl ester form.

10.3. The low-level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples without saturating the detector or leading to excessive carryover.

10.4. Initial Calibration Verification (ICV)

10.4.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. Acceptance criteria is $\pm 15\%$ for TCL analytes and $\pm 30\%$ for all other analytes. If this is not met, a new initial calibration curve is analyzed.

11. PROCEDURE

11.1. Refer to the Method 8000B section of this SOP for procedural requirements.

11.2. Extraction

11.2.1. The extraction procedure is described in SOP NC-OP-032.

11.3. Cleanup

11.3.1. The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical Sequence

11.4.1. The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.2. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new

continuing calibration run must be analyzed before proceeding with the sequence.

11.4.3. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.4. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. Gas Chromatography

11.5.1. Chromatographic conditions are listed in Table D2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the Method 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason, it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L calibration standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

12.3. A routine 10X dilution occurs on final extracts for all samples. Due to a QuantIMS limitation, the dilution factor field in QuantIMS cannot be used when a dilution is routine, because the dilution factor is automatically applied to all reference values creating reporting problems. For the herbicide analysis, the extract volume will be 10mL and an aliquot at 10X dilution will be analyzed. The final extract volume recorded on the laboratory bench sheet will be recorded as 100mL to avoid using the dilution factor field in QuantIMS.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the 8000B section of this SOP

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the 8000B section of this SOP

16. REFERENCES

16.1. Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Refer to the Method 8000B section of this SOP for modifications from the reference method.

17.2. Modifications from Previous Revision

17.2.1. The calibration procedure has been changed to require esterification of the calibration standards

17.3. TABLES

Table D1				
Standard Analyte List for Method 8151A				
Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCP	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	<u>Extraction Vol.</u>	<u>Final Vol.</u>	<u>Dilution Factor</u>
Ground water	1000 mL	10 mL	10
Low-level Soil without GPC	50g	10 mL	10
High-level soil / waste	1g	10 mL	10

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D1.

Table D2	
Instrumental Conditions for Method 8151A	
PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5 30x0.32, 0.5µm
Column 2	DB-1701 or Rtx-1701
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions must result in resolution of all analytes listed in Table D1.

The reporting limits listed in Table D1 will be achieved with these calibration levels and a 20-fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

Table D3			
LCS/Matrix Spike and Surrogate Spike levels mg/L or mg/kg¹ for Method 8151A			
	Aqueous	Soil	Waste
2,4-D	40	400	20000
Silvex	10	100	5000
2,4,5-T	10	100	5000
2,4-DB	40	400	20000
Dalapon	20	200	10000
DCAA (surrogate)	40	400	20000
Dicamba	20	200	10000
MCP	4000	40000	200000
MCPA	4000	40000	200000
Dichloroprop	40	400	2000
Pentachlorophenol	5	50	2500
Dinoseb	6	60	300

¹ LCS, MS and SS spikes are as the free acid.

Table D4						
Calibration Levels for Method 8151A and 615 (ng amount)						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
2,4 D *	0.02	0.04	0.08	0.16	0.32	0.64
2,4 DB	0.02	0.04	0.08	0.16	0.32	0.64
2,4,5 T *	0.005	0.01	0.02	0.04	0.08	0.16
2,4,5 TP (silvex) *	0.005	0.01	0.02	0.04	0.08	0.16
Dalapon	0.01	0.02	0.04	0.08	0.16	0.32
DCAA (surr)	0.02	0.04	0.08	0.16	0.32	0.64
Dicamba	0.01	0.02	0.04	0.08	0.16	0.32
Dichloroprop	0.02	0.04	0.08	0.16	0.32	0.64
Dinoseb	0.003	0.006	0.012	0.024	0.048	0.096
MCPA	2	4	8	16	32	64
MCP	2	4	8	16	32	64
Pentachlorophenol	0.0005	0.005	0.01	0.02	0.04	0.08
Picloram	0.02	0.04	0.08	0.16	0.32	0.64

* Compounds are controlling compounds in LCS and must pass criteria DCAA - surrogate

Bold levels indicate CCV

1. SCOPE AND APPLICATION

- 1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8015B is applied to the determination of the concentration and **tentative** identification of extractable petroleum (diesel range) hydrocarbon mixes in waters, wastewaters, soils, and sludges. This Appendix is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOP (NC-OP-032).
- 1.2. The analytes listed in Table E2 are routinely analyzed. Other analytes may be analyzed by this method if the quality control criteria in Section 9 and the initial demonstration of method performance in Section 13 are met. Reporting limits are also listed in Table E2. The laboratory carbon range for Ohio VAP projects is C10-C20 and C20-C32. The laboratory carbon range for BUSTR Projects is C10-C20 and C20-C34.
- 1.3. The associated LIMS method codes are HS (8015 MOD) and KI (8015B).

2. SUMMARY OF METHOD

- 2.1. This method presents conditions for the detection and identification of total petroleum hydrocarbons. Quantitation is by the external standard method. An aliquot of the prepared sample is injected into a gas chromatograph (GC) and compounds in the effluent are detected by a flame ionization detector (FID).

3. DEFINITIONS

- 3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

- 4.1. Refer to the Method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

- 5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the Method 8000B section of this SOP.
- 6.2. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

- 7.1. Refer to Section 7 of the Method 8000B section of this SOP.
- 7.2. The petroleum hydrocarbons are purchased from a chemical supplier when available. When no chemical supplier is available, the fuels are purchased from public sources. See the Standards Logbook for details on sample preparation.
- 7.3. The OVAP and BUSTR standard is a commercially prepared standard containing alkanes from C10-C34.
- 7.4. Refer to Table E3 for details of calibration standards.

8. SAMPLE PREPARATION, PRESERVATION, AND STORAGE

- 8.1. The holding time for semivolatile extracts is 40 days from extraction to analysis. Samples must be refrigerated at <6°C.

9. QUALITY CONTROL

- 9.1. Refer to Section 9 of the Method 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).
- 9.2. MS/MSD recoveries are calculated from a diesel calibration.
- 9.3. Surrogates
 - 9.3.1. Because of the nature of the TPH analysis, whereas certain petroleum mixtures can override the C9 (Nonane) surrogate.

Note: Ohio VAP rules require reanalysis when surrogate recoveries are outside of control limits. Re-extraction is required if surrogates are outside of control limits.

10. CALIBRATION AND STANDARDIZATION

Refer to Section 10 of the Method 8000B section of this SOP for general calibration requirements.

- 10.1. Recommended Instrument Conditions
 - 10.1.1. Hydrogen carrier gas - flow rate 5 - 6 mL/min
 - 10.1.2. Detector gas mixture - air hydrogen mixture in a 10:1 ratio, air 80 - 120 mL/min, hydrogen 8 - 12 mL/min
 - 10.1.3. Temperature Program - refer to Table E1, Appendix E
 - 10.1.4. Injection volume - 1 µL
- 10.2. Initial Calibration
 - 10.2.1. Analyze a five point diesel calibration standard referring to the recommended instrument conditions. The calibration concentrations are 100, 200, 500, 1000, and 2000 ng/uL. A 5000ng/uL standard may be analyzed if needed. The retention time window of C10-C32 must be used for the Diesel calibration. The low-level standard must be at or below the reporting limit. The other standards define the working range of the detector.
 - 10.2.2. For Ohio VAP and BUSTR projects, the laboratory analyzes a five-point calibration for the carbon range C10-C20. The concentrations are 60, 120, 240, 600 and 1200 ug/mL. In addition, a five point of the carbon range C20-C34 is also analyzed. The concentration ranges are 80, 160, 320, 800, and 1600 ug/mL. These standards serve as the marker solution and are used to determine the retention time window for each carbon range.
- 10.3. Initial Calibration Verification (ICV)
 - 10.3.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. Acceptance criteria is $\pm 15\%$. If this is not met, a new initial calibration curve is analyzed.
- 10.4. Continuing Calibration
 - 10.4.1. Refer to Section 10 of the Method 8000B section of this SOP for general calibration requirements.
 - 10.4.2. A mid-range standard of diesel, C10-20, and C20-34 is used, as appropriate, for the CCV. The acceptance criteria is $\pm 15\%$. This marker solution must be analyzed at the beginning of each sequence.

11. PROCEDURE

- 11.1. Refer to the Method 8000B section of this SOP for procedural requirements.
- 11.2. Extraction
 - 11.2.1. The extraction procedure is described in SOP NC-OP-032.
- 11.3. Analytical Sequence – Refer to Section 11 in the Method 8000B Section of this SOP.
- 11.4. Petroleum Hydrocarbon Identification and/or Fingerprinting
 - 11.4.1. To identify the type of petroleum hydrocarbon, compare the chromatographic peak pattern to the patterns of known petroleum hydrocarbons analyzed under identical chromatographic conditions. Samples are quantified against diesel, but fingerprinting may be done when client requested.
 - 11.4.2. Positive matching may not be possible, even using site-specific hydrocarbons. Degradation of the pattern can occur during environmental exposure of the fuel. See Table 2 for possible fingerprints.
 - 11.4.3. Samples are quantified against the initial calibration of diesel or DRO on a single column.
 - 11.4.4. The total height or area of the hydrocarbon is determined in the same manner used for the hydrocarbon standard.
 - 11.4.5. If the amount of sample injected into the GC exceeds the working range of the calibration curve, an appropriate dilution is performed before reanalysis.
- 12. DATA ANALYSIS AND CALCULATIONS**
 - 12.1. Refer to the Method 8000B section of this SOP for identification and quantitation of single component analytes.
 - 12.2. Surrogate recovery results are calculated and reported for Nonane (C-9). The surrogate must be within QC criteria. Corrective action is only necessary if Nonane (C-9) is outside of acceptance limits.
- 13. METHOD PERFORMANCE**
 - 13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.
- 14. POLLUTION PREVENTION**
 - 14.1. Refer to Section 14 of the 8000B section of this SOP.
- 15. WASTE MANAGEMENT**
 - 15.1. Refer to Section 15 of the 8000B section of this SOP.
- 16. REFERENCES**
 - 16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA
 - 16.2. Related SOP
 - 16.2.1. NC-OP-032, Extraction of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, 8150, 8151, and 600 Series Methods

Table E1

Suggested GC Temperature Program for TPH Analysis

Initial Temperature	40°C
Initial Hold Time	4 minutes
Temperature Program	10°C/minute
Final Temperature	280°C
Final Hold Time	10 minutes

Table E2
Reporting Limits for TPH Analysis

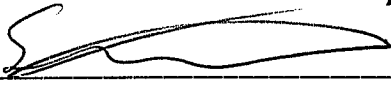
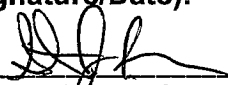



Analyte	Reporting Limits		
	Water (mg/L)	Solids (mg/kg)	Waste Dilution (mg/kg)
TPH (as Diesel) or DRO	100	3.3	200
C10-C20 (OVAP & BUSTR)	60	2.0	
C20-C34 (OVAP & BUSTR)	80	2.3	
Fingerprint Compounds ¹			
Mineral Spirits	Kerosene	Motor Oil	
Hydraulic Oil	Jet Fuel	Stoddard Solvent	
DRO Spiking Solution			
Decane	Dodecane	Tetradecane	
Hexadecane	Octobecane	Eicosane	
Docosane	Tetracosane	Hexacosane	
Octacosane			

¹ This list represents most of the common petroleum hydrocarbons. The list may be expanded to include other petroleum hydrocarbons.

Table E3					
Calibration Levels for Method 8015B (ng/L)					
	Level 1	Level 2	Level 3	Level 4	Level 5
TPH (as Diesel)	100	200	500	1000	2000
C10-C20 (OVAP & BUSTR)	60	120	240	600	1200
C20-C34 (OVAP & BUSTR)	80	160	320	800	1600

**Title: TOXICITY CHARACTERISTIC LEACHING PROCEDURE
AND SYNTHETIC PRECIPITATION
LEACHING PROCEDURE**

[SW846 Method 1311]

Approvals (Signature/Date):			
	3-18-08		3/18/08
Technology Specialist	Date	Health & Safety Coordinator	Date
	3/19/08		3/20/08
Quality Assurance Manager	Date	Laboratory Director	Date
	3/19/08		
Technical Director	Date		

This SOP was previously identified as SOP CORP-IP-0004NC, Rev 1.2, dated 11/11/04

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1. SCOPE AND APPLICATION

1.1. This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW846 Method 1311. The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC is one of four criteria in 40 CFR Part 261 to determine whether a solid waste is classified as a hazardous waste. The other three are corrosivity, reactivity, and ignitability. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions that were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2. The specific list of TC analytes and regulatory limits may be found in Appendix A.

Note: The list in Appendix A does not include the December 1994 EPA rule for Universal Treatment Standards for Land Disposal Restrictions. Those requirements include 216 specific metallic and organic compounds and, in some cases, lower detection limit requirements (see 40 CFR 268.40). TCLP leachates are part of the new Universal Treatment Standards, but the conventional analytical methods will not necessarily meet the new regulatory limits. Consult with the client and with TestAmerica Technical Specialists before establishing the instrumental methods for these regulations.

1.3. This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Appendix A. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.

1.4. The procedure is applicable to liquid, solid, and multiphase wastes.

1.5. The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

- 1.6. The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.
- 1.7. If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.
- 1.8. If an analysis of any one of the liquid fractions of the procedure leachate indicates that a regulated compound is present at such a high concentration that, even after accounting for dilution from the other fractions of the leachate, the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it may not be necessary to analyze the remaining fractions of the leachate. However, the remaining analyses should not be terminated without the approval of the client.
- 1.9. Volatile organic analysis of the leachate obtained using a bottle extraction, normally used for extractable organics and metals, can be used to demonstrate that a waste is hazardous, but only the ZHE option can be used to demonstrate that the concentration of volatile organic compounds is below regulatory limits due to potential analyte loss into the headspace during the bottle extraction.

2. SUMMARY OF METHOD

- 2.1. For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP leachate.
- 2.2. For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis or recombination with the leachate. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. For TCLP, the extraction fluid employed for extraction of non-volatile analytes is a function of the alkalinity of the solid phase of the waste. For SPLP, the extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater the extraction fluid employed is a pH 4.2 solution. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals, and/or, b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following extraction, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 μm fiber filter.

- 2.3. If compatible (i.e., multiple phases will not form on combination), the initial liquid filtrate of the waste is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3. DEFINITIONS

- 3.1. “Leachate” is used to refer to the solutions generated from these procedures (TCLP, SPLP, deionized water leach).
- 3.2. “Wet Solids” is that fraction of a waste sample from which no liquid may be forced out by pressure filtration.

4. INTERFERENCES

- 4.1. Oily wastes may present unusual filtration and drying problems. If requested by the client and as recommended by EPA (see Figure 3), oily wastes can be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter. Alternately, the oil may be subjected to pressure filtration. The portion that passes through the filter will be prepared and analyzed separately as an organic waste. The “wet solid” portion that remains behind on the filter will be subjected to leaching--prepared and analyzed. The results will then be mathematically combined.
- 4.2. Wastes containing free organic liquids (e.g., oil, paint thinner, fuel) usually require dilution prior to analysis to address the matrix interferences. In most instances this results in reporting limits elevated above the TCLP regulatory limits.
- 4.3. Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks as described in Section 9 and the individual determinative SOPs.
- 4.4. Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.
- 4.5. Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.
- 4.6. Overexposure of the sample to the environment will result in the loss of volatile components.

- 4.7. Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.
- 5.2. Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized. CAUTION: Do not open equipment that has been pressurized until it has returned to ambient pressure.
- 5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Glacial Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m ³ - TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm- TWA 4 ppm- STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methylene Chloride	Carcinogen Irritant	25 ppm- TWA 125 ppm- STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 mg/m ³ - Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

- 5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.
- 5.5. A rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene overwraps of the glass jar, may be necessary. Turning the jar or bottle sideways rather than tumbling end over end may also reduce the chance of breakage. If sideways tumbling is used, note this change in the logbook comment section. Guards must be placed in front of any rotating equipment.
- 5.6. Secure tumbler and extraction apparatus before starting rotary agitation apparatus.
- 5.7. During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure.
- 5.8. Any cyanide containing waste or soil may result in the formation of hydrogen cyanide gas when exposed to acidic conditions. SPLP Fluid #3 reagent water must be used for these samples. **NOTE: Do not use an acidic SPLP fluid due to the potential release of hydrogen cyanide gas.**
- 5.9. Exposure to hazardous chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.10. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 5.11. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and Laboratory Supervisor.
- 5.12. Due to the potential for ignition, flammability or production of noxious fumes, do not attempt to dry non-aqueous liquid samples in an oven. Use extended drying in a ventilation hood.

6. EQUIPMENT AND SUPPLIES

- 6.1. Extraction vessels
 - 6.1.1. For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Figure 2)
 - 6.1.2. For metals - either borosilicate glass jars (2.5 L, with Teflon lid inserts) or 2.5 L HDPE (Nalgene or equivalent) bottles may be used
 - 6.1.3. For non-volatile organics - only borosilicate glass may be used
- 6.2. Vacuum filtration apparatus and stainless steel pressure filtration apparatus (142 mm diameter), capable of 0 - 50 psi
- 6.3. Borosilicate glass fiber filters, 0.6 - 0.8 μm (Whatman GF/F 14.2 cm, 9.0 cm, 4.7 cm, 0.7 μm or equivalent). When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use, or purchase pre-washed filters. Glass fiber filters are fragile and should be handled with care.
- 6.4. Rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Figure 1). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.
- 6.5. ZHE Extract Collection Device: Gas-tight syringes, 50 or 100 mL capacity, Hamilton 0158330 or equivalent
- 6.6. Top loading balance, capable of 0 - 4000 \pm 0.01g (all measurements are to be within \pm 0.1 grams)
- 6.7. pH meter and probe capable of reading to the nearest 0.01 unit, and with automatic temperature compensation
- 6.8. Magnetic stirrer/hotplate and stirring bars
- 6.9. VOA vials, 40 mL, with caps and septa
- 6.10. Glass bottles, 1 liter, with Teflon lid-inserts
- 6.11. Nalgene plastic bottles or equivalent, 1 liter
- 6.12. Miscellaneous laboratory glassware and equipment

7. REAGENTS AND STANDARDS

- 7.1. Reagent water for non-volatile constituents must be produced by a Millipore DI system or equivalent. For volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through activated carbon). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
- 7.2. Hydrochloric acid, 1 N: Carefully add 83 mL concentrated reagent grade HCl to 800 mL reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well.
- 7.3. Sodium hydroxide, 1 N: Carefully add 40 g reagent grade NaOH pellets to 800 mL reagent water, stir until the pellets are completely dissolved, cool and dilute to 1 liter with reagent water.

CAUTION: Heat is generated during this process.

- 7.4. Acetic acid, glacial: concentrated, reagent grade liquid (HOAc)
- 7.6. pH calibration solutions: buffered to a pH of 4, 7, and 10. Commercially available.
- 7.5. TCLP Leaching Fluids

7.5.1. General Comments

- 7.5.1.1. The pH of both solutions listed below should be monitored daily and the pH probes are to be calibrated prior to use.
 - 7.5.1.2. The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.
 - 7.5.1.3. Additional volumes of extraction fluids listed below may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.
 - 7.5.1.4. At the end of the day, all remaining buffer solutions must be properly discarded.
- 7.5.2. TCLP Fluid #1: Carefully add 5.7 mL glacial acetic acid and 64.3 mL of 1 N NaOH to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. For 8 L of fluid use

45.6 mL glacial acetic acid and 514 mL 1N NaOH, dilute to 8 L with reagent water. When correctly prepared, the pH of this solution is 4.93 ± 0.05 . The density of TCLP fluid #1 is 0.997 g/mL.

- 7.5.3. TCLP Fluid #2: Carefully add 5.7 mL glacial acetic acid to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. For 8 L of fluid use 45.6 mL glacial acetic acid, dilute to 8 L with reagent water. When correctly prepared, the pH of this solution is 2.88 ± 0.05 . The density of TCLP fluid #2 is 0.997 g/mL.
- 7.6. Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL reagent water. Cap and shake to mix well.
- 7.7. Sulfuric acid / nitric acid (60/40 weight percent mixture) $\text{H}_2\text{SO}_4/\text{HNO}_3$. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.
- 7.8. SPLP Leaching fluids
- 7.8.1. SPLP solutions are unbuffered and exact pH may not be attained. The pH of TCLP and SPLP fluids should be checked prior to use. If not within specifications, the fluid should be discarded and fresh fluid prepared.
- 7.8.2. SPLP fluid #1: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 4.20 ± 0.05 . This fluid is used for soils from a site that is east of the Mississippi River and for wastes and waste waters.
- 7.8.3. SPLP fluid #2: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05 . This fluid is used for soils from a site that is west of the Mississippi River.
- 7.8.4. SPLP fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.
- 7.9. Methanol and methylene chloride - used to aid in cleaning oil contaminated equipment.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Samples being analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation. Undamaged glass containers should be washed according to SOP NC-QA-0014.

- 8.2. Samples being analyzed for metals only can be collected in either glass or polyethylene containers.
- 8.3. When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon lined septum capped vials with minimal headspace and stored at $4 \pm 2^{\circ}\text{C}$). Samples should be opened only immediately prior to extraction.
- 8.4. Samples should be refrigerated to $4 \pm 2^{\circ}\text{C}$ unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.
- 8.5. The minimum TCLP sample collection size is determined by the physical state or states of the waste and the analytes of concern. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than 50% solids), minimum requirements are 2 - 32 oz jars for semi-volatile organic analysis and metals, and 2 - 8 oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required. If matrix spike or duplicate control samples are requested, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible.
- 8.6. TCLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate shall not be acidified and the leachate shall be analyzed as soon as possible. All other leachates should be stored under refrigeration ($4 \pm 2^{\circ}\text{C}$) until analyzed. ZHE leachates must be stored in VOA vials filled to eliminate all headspace.
- 8.7. Samples are subject to appropriate treatment within the following time periods.

Table 1
Holding Times (days)

Parameter	Collection to Start of TCLP Leach	End of TCLP Tumble to Preparation	Start of TCLP Leach or Semi- volatile Prep Extraction to Analysis	Total Elapsed Time
Volatiles	14	N/A	14	28
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Other Metals	180	N/A	180	360

NOTE: The initial holding time is measured from date of collection to date TCLP extraction started. (This should be the TCLP extraction date in QuantIms.) Semi-volatile method prep holding time is measured from the day tumbling is complete to the start of method extraction. Subsequent analysis holding times are measured from the date extraction (TCLP or method prep) starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit. The Total Elapsed Time is to be used as guidance. If preps are initiated at the last possible moment of a holding time, the elapsed times may be exceeded.

9. QUALITY CONTROL

- 9.1. Quality Control Batch (QC Batch) - QA-003 defines a QC Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same procedures, reagents and standards within the same time period. The same lot of reagents must be used within a batch. A minimum of one TCLP extraction blank (Method Blank), one Laboratory Control Sample (LCS), one Matrix Spike (MS), and one Matrix Spike Duplicate (MSD) will be prepared with each TCLP leachate batch.
- 9.2. TCLP Extraction Blanks - A minimum of one blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples extracted in a particular vessel type. The blanks are generated in the same way as the samples (i.e., blanks will be tumbled and filtered with the samples). If particle size reduction was performed on any sample in the batch, an equipment blank will be generated by passing blank fluid through the particle reduction apparatus. ZHE Extraction vessels will be uniquely numbered. Consult the TestAmerica QC Program and the individual analysis SOPs for blank acceptance criteria.
- 9.3. Laboratory Control Sample (LCS) - A LCS is required with each batch of 20 or fewer samples. The LCS shall be generated after a batch of TCLP leachates have been

generated (i.e., at the time of the preparative digestion or extraction) by spiking an aliquot of the appropriate extraction fluid used for that batch or reagent water. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.4. Matrix Spike (MS/MSD) - Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. A MS/MSD pair are required with each batch of 20 or fewer samples.

9.4.1. Matrix spikes are to be added after filtration of the TCLP leachate. Spikes are not to be added prior to the TCLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.

9.4.2. Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.5. Corrective Actions

9.5.1. Consult the TestAmerica QC Program and individual analysis SOPs for corrective action for blanks and LCS

9.5.2. Method of Standard Additions (MSA) shall be used for mercury if all of the following conditions are met:

- Recovery of the analyte in matrix spike is not at least 50%,
- The concentration of the analyte does not exceed the regulatory level, and
- The concentration of the analyte measured in the sample is within 20% of the appropriate regulatory level.

If the matrix spike recovery is 5% or less due to dilution or matrix interference, contact the project manager and client for guidance. The client should also be contacted prior to initiation of any MSA steps. Refer to the individual analysis SOPs for details on how to perform MSA analysis.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to appropriate analysis SOPs.

11. PROCEDURE

11.1. General Comments

11.1.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented on a Nonconformance Memo kept in the project file and described in the final report. The variation must be approved by a Project Manager, Technical Specialist, and QA Manager. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.1.2. All masses should be recorded to the nearest 0.1 g.

11.2. Preliminary Sample Evaluations (Refer to Flow Chart #1, Appendix D)

11.2.1. Determine the total volume of TCLP leachate (solid phase leachate plus liquid filtrate) that needs to be generated for analysis according to the following:

Table 2
Recommended TCLP Leachate Volume

Analysis	TCLP Required Volume (mL)	SPLP Required Volume (mL)
Volatiles	3 x 40	3 x 40
Semi-volatiles	500	1000
Pesticides	500	1000
Herbicides	500	1000
Metals	300	300

11.2.1.1. For TCLP and SPLP samples used for matrix spike and matrix spike duplicate analysis, two to three times the listed volumes are required.

11.2.2. Sample Description (determine sample matrix)

11.2.2.1. Solid - If the waste will obviously yield no free liquid when subjected to pressure filtration, then proceed to Section 11.2.5 or 11.4 (Bottle Extraction Procedure or ZHE Procedure).

11.2.2.2. Liquid - If the sample is a monophasic liquid, proceed to Section 11.2.3 (Percent Solid Determination).

11.2.2.3. Multiphasic – The sample has discernible layers (liquid/liquid or liquid/solid). If more than one container of multi-phasic materials is received from the field, each container might show different amounts of each phase. Consult client to determine sample selection alternatives (composite all sample containers, select one, resample, etc.) if this occurs.

11.2.3. Solids Determination

11.2.3.1. Determine Type of Filtration Apparatus and Process

11.2.3.1.1. Percent Solids and ZHE Extractions - The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 11.4 (ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with Section 11.2.3.2. The aliquot of sample used here cannot be used again for the ZHE extraction.

11.2.3.1.2. If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (expected to be < 0.5%) , vacuum filtration should be used initially. Proceed to determination of percent dry solids (Section 11.2.3.2)

11.2.3.1.3. If the sample is viscous (sludge, oil, or is expected to have solids content > 0.5%), use pressure filtration. Proceed to determination of wet solids (Section 11.2.3.3).

11.2.3.2. Determination of percent dry solids

11.2.3.2.1. Measure and record the weight of the filter. Load the filter into the filter holder and assemble vacuum filter apparatus.

11.2.3.2.2. Homogenize the waste, then transfer 100 g subsample to a glass beaker. Record the sample weight in the percent dry solids section of the logbook.

11.2.3.2.3. Turn on vacuum source. Transfer the sample to the vacuum filtration device attempting to spread the waste sample evenly over the surface of the filter. Be sure to transfer all

particulates from the beaker to the filter. Use a reagent water rinse if necessary.

- 11.2.3.2.4. Once all liquid has been pulled through the filter, remove the filter with the wet solids from the vacuum filtration apparatus.
- 11.2.3.2.5. Dry the filter and solid phase at 100 ± 20 ° C for approximately 15 minutes.
- 11.2.3.2.6. Remove the filter from the oven, and allow to cool in a desiccator.
- 11.2.3.2.7. Weigh and record the dry weight of filter + particulates.
- 11.2.3.2.8. Calculate and record the percent dry solids.
- 11.2.3.2.9. If the percent dry solids is $\geq 0.5\%$, repeat the drying step. Weigh and record the second filter + particulates dry weight. If the two weighings do not agree within 1%, perform additional drying and weighing until successive weighings agree within 1%.
- 11.2.3.2.10. If the dry solids result is $\geq 0.5\%$, proceed to Section 11.2.3.3 using a fresh wet portion of the multiphase waste.
- 11.2.3.2.11. If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary. Filter sufficient sample with either the pressure filtration system or ZHE system as described in Sections 11.3 and 11.4. The filtrate is the TCLP leachate.

11.2.3.3. Determination of wet solids

- 11.2.3.3.1. Assemble the pressure filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 μ m filter membrane).
- 11.2.3.3.2. Homogenize the waste, transfer a minimum of a 100 mL subsample to the glass beaker. Measure and record the gross weight (logbook Column A).
- 11.2.3.3.3. Measure and record the tare weight of the filtrate collection bottle (logbook Column D).

- 11.2.3.3.4. Transfer the sample to the filtration device attempting to spread the waste sample evenly over the surface of the filter. Measure and record the tare weight of the empty glass beaker and any residual sample (logbook Column B).
- 11.2.3.3.5. Calculate and record the net weight of sample used for testing (logbook Column C).
- 11.2.3.3.6. Slowly apply gentle pressure of 10 psi to the filtration apparatus. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter during a two-minute period.
- 11.2.3.3.7. If necessary, repeat previous step by increasing the pressure in 10 psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a two-minute period.

Note: Some samples will contain liquid material that does not filter (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils, or any wastes which do not pass through the filter, are classified by the method as a solid.
- 11.2.3.3.8. Remove the filtrate collection bottle, weigh and record the gross weight (logbook Column E).
- 11.2.3.3.9. Calculate and record the net weight of filtrate (logbook Column F). This result will be used in the percent solids calculation.
- 11.2.3.3.10. To determine the amount of filtrate, place the exact same type and size container as the filtrate container next to the filtrate. Add water to the exact level as the filtrate container to the empty container. Transfer the water to a graduated cylinder and record the volume. This step will reduce the amount of contamination, which may exist from transferring the filtrate to a graduated cylinder.
- 11.2.3.3.11. Retain the filtrate for possible recombination with the leachate in Section 11.3.7. Retain the filter and wet solids for the leaching in Section 11.3.

- 11.2.3.3.12. For multiphase sample preparations, calculate the total weight of wet solids and record the result in logbook Column G.

11.2.4. Particle-size Reduction for Fluid Selection

- 11.2.4.1. The subsample used for fluid selection must consist of particles less than approximately 1 mm in diameter (versus the less than 1 cm requirement for the material used for the actual extraction). The method requires a smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full extraction. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.
- 11.2.4.2. Surface area exclusion - size reduction is not required if the sample surface area is greater than or equal to 3.1 cm² per gram.
- 11.2.4.3. If the sample contains particles greater than approximately 1 mm in diameter, crush, cut, or grind the solids to the required size.
- 11.2.4.4. Consult a supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick).

11.2.5. Determination of Appropriate Extraction Fluid

- 11.2.5.1. If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or nonvolatile organic compounds, the type of leaching solution must be determined.
- 11.2.5.2. Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.
- 11.2.5.3. For SPLP, refer to Section 7.8 for fluid selection. Record the fluid type in the logbook.
- 11.2.5.4. The TCLP leaching fluid for all volatiles is TCLP Fluid #1.
- 11.2.5.5. TCLP leach fluid determination for non-volatile analytes
- 11.2.5.5.1. Calibrate the pH meter with fresh buffer solution in accordance with the pH SOP.

11.2.5.5.2. Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a glass or plastic container, and record in the logbook. Note: If sample quantity is limited, consult supervisor or manager.

Note: Many multiphase samples have limited solids quantity . In these instances, use a 5 g aliquot of the whole sample. Document this difference in the logbook comment section.

11.2.5.5.3. Add 96.5 ± 1.0 mL of reagent water, add magnetic stir bar, cover with a watchglass, and stir for 5 minutes.

11.2.5.5.4. Measure and record the pre-test sample pH in the logbook.

Note: To avoid damaging a glass pH probe when organic liquid is present, use narrow range pH indicator paper or an ISFET pH meter.

11.2.5.5.5. If the pH is less than or equal to 5.0, use TCLP Fluid #1.

11.2.5.5.6. If the fluid pH is greater than 5.0, add 3.5 mL 1 N HCl. Slurry the sample briefly. Insert therm into room temp DI water in one vial in each pre-test sample group to monitor the temperature. All samples in the group must be heated at the same time in order for the temperature of the one monitored sample to represent the others. Heat to $50 \pm 2^{\circ}\text{C}$ and maintain for ten minutes.

Note: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

11.2.5.5.7. Cool to room temperature.

11.2.5.5.8. Measure and record the pH immediately after the sample has reached room temperature.

11.2.5.5.8.1. If the pH is less than or equal to 5.0, use TCLP Fluid #1. Record the buffer in the logbook.

11.2.5.5.8.2. If the pH is greater than 5.0, use TCLP Fluid #2. Record the buffer in the logbook.

11.2.6. For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals proceed to Section 11.3.

11.2.7. For samples requiring analysis for volatile organics (ZHE), proceed to Section 11.4.

11.3. Bottle Extraction Procedure: Non-Volatile Constituents: Semi-Volatiles, Pesticides, Herbicides, Metals (Refer to Flow Chart #2, Appendix D)

11.3.1. Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e., capable of passing through a 9.5 mm, 0.375 inch, standard sieve). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm² per gram. If particle size reduction was required, record this in comments column in logbook.

11.3.1.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Determination of particle size reduction tools should take into account the requested analytes (e.g. avoid chromium steel tools when TCLP metals have been requested). Bricks, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction (contact PA or PM). Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

11.3.2. Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 11.2.1.

11.3.3. Use 100 g of solid unless sample quantity is limited. If limited sample, divide the total volume of solid phase leachate required by 20 to determine the minimum mass of solid phase required for leaching. Round this mass UP to the nearest 5g. Client must be notified if less than 100 g of solid material is used.

Note: Solid phase material is often in limited quantity from multiphase samples. Generally all the *solid* phase material and the filter from Section 11.2.3.3.11 are transferred to the leaching bottle.

- 11.3.4. All non-ZHE extraction vessels should be uniquely numbered. If breakage occurs, replacement vessels must be numbered with the original vessel identification number prior to use. Breakage must be noted in the logbook comments identifying the affected sample and vessel number.
- 11.3.5. Weigh the required mass of solid phase into an appropriate extraction vessel (plastic for metals only, glass for all others) and **slowly** add 20 times its mass of appropriate leaching fluid (e.g., 100 g of sample would require 2000 mL of leaching fluid). Record the weight of the sample aliquoted for the extraction. Record the volume of extraction fluid added in the logbook if other than 2000 mL. Record the pH.
- 11.3.6. Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room should be $23 \pm 2^{\circ}\text{C}$. Record the rotary agitator I.D. and the date and time extraction is started and completed in the logbook.
- Note:** As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.
- 11.3.7. After tumbling in the rotary agitator is completed, remove the bottle and allow the solids to settle. Record the date and time the extraction is completed in the logbook. If sample was multiphase with an initial filtrate, drop a few drops of the filtrate (with a disposable glass pipette) into the extraction bottle and observe whether the filtrate is insoluble or forms a precipitate with the leachate. If so then the filtrate is not compatible with the leachate and must be bottled and analyzed separately. The results are normally mathematically recombined (Section 12.1.2). If the filtrate is compatible with the leachate (i.e., completely soluble) then pour the entire filtrate into the leachate bottle, recap and mix. Proceed with the leachate filtration step in the next section.
- 11.3.8. Filter the sample using pressure filtration by filtering through a new glass fiber filter. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). The entire sample need not be filtered; however, sufficient volume should be generated to support the required analyses.
- 11.3.9. Measure the pH of the TCLP leachate and record in the logbook. (Use narrow range pH paper or ISFET pH meter to measure the pH of oily samples as a glass pH probe may be damaged.)

11.3.10. Prepare sufficient volume for MS/MSD quality control testing. Refer to the appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.

11.3.11. Immediately preserve the leachate as follows:

Metals pH < 2 w/ HNO₃ for aqueous filtrates and leachates
(do not acidify oils and other non-aqueous liquids)

All others Refrigerate to 4 ± 2 °C

Note: Refer to Section 8.6 if precipitation occurs upon preservation.

11.3.12. Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis. For multiphase samples requiring mathematical recombination provide copies of the TCLP preparation logbook sheets to the sample preparation and analysis groups. Most mathematically recombined samples will require data entry for the filtrate and leachate portions as well as for the mathematically recombined results. Contact the Project Manager to ensure the proper sample login is completed.

11.4. ZHE Extraction Procedure: Volatile Constituents (Refer to Flow Chart #3, Appendix D)

11.4.1. Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, herbicides and semi-volatile organics).

11.4.2. Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere any longer than necessary.

11.4.3. Install new O-rings and adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

11.4.4. If the preliminary evaluations indicated the need for particle size reduction, homogenize the waste, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size as measured with a ruler (Do NOT sieve the sample). Size

reduction is not required if the sample surface area is greater than or equal to 3.1 cm² per gram. If particle size reduction was required, record this in the comments column of the logbook.

Note: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4°C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate. Also see Section 11.3.1.

11.4.4.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction (contact PM).

11.4.5. Homogenize and transfer an appropriate size subsample of the waste into the ZHE and record the mass in the logbook.

11.4.5.1. For wastes that are solid, a 25 g sample is used.

11.4.5.2. For wastes containing < 0.5% solids, the liquid portion of the waste, after filtration, is defined as the TCLP leachate. Filter enough of the sample to support all of the volatile analyses required.

11.4.5.3. If the sample has ≥ 0.5% solids and has non-volatile TCLP/SPLP requested, the appropriate sample size should be estimated based on the wet solids content determined in Section 11.2.3.3. If ZHE only, use visual wet solids estimate to sample subaliquot.

Note: For wastes containing greater than 0.5% wet or dry solids, the “solids” value from the ZHE filtration process may be used to determine the volume of fluid to load into the ZHE. This approach is recommended since the solids value from Section 11.2.3.3 may differ from the ZHE filtration solids due to sample variability or differences in the filtration apparatus.

11.4.6. Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

11.4.7. Place the ZHE in a vertical position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

- 11.4.8. If the waste is solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 11.4.13.
- 11.4.9. If this is a mutliphase sample, carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.
- 11.4.10. Assemble a syringe and place the plunger in all the way. Attach the pre-weighed syringe to the liquid inlet/outlet valve and open the valve. Record the tare weight of the collection syringe in Column D of the logbook. .
- 11.4.11. Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a two-minute period.

Note: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe, weigh, record weight in Column E and filtrate volume in the logbook. Return to Section 11.4.10.

- 11.4.12. Repeat previous step increasing the pressure in 10 PSI increments until 50 psi is reached and no significant liquid has passed in a 2 minute period. Close the valve and discontinue gas pressure. Remove the collection device and record the total weight of the collection device with filtrate in column E and filtrate volume in the logbook. Transfer the filtrate to VOA vials and label appropriately. Calculate the weight of filtrate collected and record in Column F in the logbook.

Note: If the original waste contained less than 0.5% solids (Section 11.2.3.2), this filtrate is defined as the TCLP leachate and you may proceed to Section 11.4.22. Otherwise, save the vials by storing at 4°C under minimal headspace conditions, for recombination as in Section 11.4.21.

The material remaining in the ZHE is defined to be the “solid” phase. Calculate the weight of the solid phase and record in Column G of the logbook by subtracting the weight of the filtrate from the weight of the sample.

- 11.4.13. Determine the amount of buffer to use. Solid samples use 500 mL of leach fluid (20 X 25 g). For multiphase samples use the wet solids (Column G) amount and multiply by 20. Record the leach fluid volume in Column H of the logbook.

Note: The TCLP ZHE prep uses only TCLP fluid #1; the SPLP ZHE prep uses only SPLP fluid #3.

- 11.4.14. Load the fluid transfer reservoir with an excess of Fluid #1 and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.
- 11.4.15. Attach the transfer line to the liquid inlet/outlet valve and open the valve. Carefully pump the required volume into the ZHE and close the valve. Disconnect the transfer line.
- 11.4.16. Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) two or three times. Reposition the ZHE in the vertical position.
- 11.4.17. Pressurize the ZHE to 5-10 psi. If the ZHE appears to be leaking, follow the corrective action protocols recommended by the manufacturer and repeat the analysis.
- 11.4.18. Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.
- 11.4.19. Repressurize the ZHE to 5-10 psi and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours. Room temperature should be 23 ± 2 °C. The room temperature is recorded using a continuous temperature monitor.
- 11.4.20. Confirm that the pressure of 5-10 psi was maintained throughout the leaching. If it was NOT maintained, return to Section 11.4 and repeat the leachate with a new aliquot of sample.
- 11.4.21. If there is an initial liquid filtrate (Sec 11.4.12) determine if it is compatible with the leachate if the filtrate has not been previously tested (Sec. 11.3.7).
 - 11.4.21.1. Remove the plunger from the syringe and attach the barrel to the ZHE vessel. Open the outlet valve and pressurize as necessary to transfer about 1 mL of leachate into the syringe. Close the outlet valve.
 - 11.4.21.2. With a glass pipette transfer a few drops of initial filtrate into the open syringe barrel. Formation of separate layers or a precipitate indicates the filtrate and leachate are not compatible. Bottle the filtrate for separate preparation and analysis. The results are normally mathematically recombined.
 - 11.4.21.3. If the filtrate is compatible gently pour the remainder of the filtrate into the syringe barrel. Install the plunger. Bleed any pressure in the

ZHE piston. Open the inlet/outlet valve and depress the syringe plunger to inject the filtrate into the ZHE vessel. Do not inject the air bubble (if present) from the syringe.

11.4.21.4. Close the valve and rotate a few times to mix. Proceed with leachate filtration as described in the next section.

11.4.22. Attach an empty syringe to the outlet valve. Open the valve and pressurize the piston to expel the leachate from the ZHE vessel. Following collection, store the TCLP leachate in 2 or 3 40-mL VOA vials with minimal headspace at 4 ± 2 °C and prepare for analysis as soon as possible using the appropriate organic analysis procedure (see Section 16.3).

11.4.23. If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.1.2. Provide copies of the TCLP preparation logbook sheets to the sample preparation and analysis groups. Most mathematically recombined samples will require data entry for the filtrate and leachate portions as well as for the mathematically recombined results. Contact the project manager to ensure the proper sample login is completed.

11.4.24. ZHE Vessel Cleaning

11.4.24.1. Disassemble the vessel.

11.4.24.2. Clean all parts (vessel, lid, bottom, piston, and metal filters) with soapy water.

11.4.24.3. Rinse all parts with tap water followed by D.I. water.

11.4.24.4. Discard all used gaskets.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculations

12.1.1. Calculation of weight of extraction fluid to use:

$$\text{Volume of extraction fluid} = 20 \times \text{weight of wet solids to be extracted}$$

12.1.2. Mathematical recombination of analytical results:

$$\text{Final Analyte Concentration} = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

Where,

V_1 = total volume of the initial filtrate phase (L).

C_1 = analyte concentration in initial filtrate phase (mg/L).

V_2 = volume of the theoretical solid phase leachate (L).

C_2 = analyte concentration in solid phase leachate (mg/L),

12.2. Reporting Requirements

12.2.1. Follow these reporting conventions for multi-phase samples.

- 12.2.1.1. If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.
- 12.2.1.2. If both phases are “ND” (not detected) the recombined result is “ND,” and the reporting limit is calculated from the reporting limit for each phase.
- 12.2.1.3. If one phase is “ND” and the other phase has a positive result, use the zero for the “ND” phase and the positive value for the other phase to calculate the combined result. This will produce a minimum known concentration. Alternatively, at client request, the maximum possible concentration can be calculated by using the reporting limit for the “ND” phase rather than zero. The combined reporting limit is based on the reporting limit for both phases

12.2.2. Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

12.2.3. For limits and significant figures, consult the appropriate analytical methods (Section 16.3).

12.2.4. Anomalies - all anomalies observed during the leach procedure must be noted on the worksheet or an NCM form. Some examples of such anomalies are:

- 12.2.4.1. Sample was monolithic - particle size reduction not possible due to nature of matrix.

12.2.4.2. Insufficient sample - less than the required 100 g minimum was available.

12.3. Review Requirements

12.3.1. Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation was properly documented in an NCM.

12.3.2. If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

$$Total \geq 20 \times TCLP$$

Note: Assumes the sample is 100% Solids.

12.3.3. Total constituent analysis results can be used to demonstrate the TCLP protocol is unnecessary. In performing a TCLP analysis, there is a 20:1 dilution of the original sample with the leaching solution. Thus, if the “total constituent” result is less than 20 times the TC level, it is impossible for the leachate to “fail” and the TCLP does not need to be performed. For example, the TC level for lead is 5.0 mg/L (ppm). Therefore, if a sample of lead-contaminated soil contains less than 100 ppm total lead, a TCLP test need not be run to demonstrate that lead is less than the TCLP limit.

13. METHOD PERFORMANCE

13.1. Refer to individual analysis SOPs.

13.2. Training Qualification:

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the

potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Acidic waste from sample extract. This waste is collected in the laboratory in a designated container identified as "Acid Waste".

15.2.2. Buffer solutions. This waste can be poured down the drain with copious amounts of water.

15.2.3. Solid waste from sample extract, solid sample waste and used filter paper from the sample filtration step. This waste is disposed of in a designated container identified as "Solid Waste."

15.2.4. Flammable solvent waste and remaining TCLP extracts. This waste is disposed of in a flammable liquid solvent container identified as "Mixed Flammable Solvent Waste".

15.2.5. Glassware contaminated with acidic sample residue. Broken or unusable glassware is disposed of in a designated container identified as "Solid Waste." Glassware used in this test method is washed per the Glassware Washing SOP.

16. REFERENCES

16.1. Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I.

16.2. Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1994, SW-846 Update II.

16.3. Related Documents

16.3.1. Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990

16.3.2. Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990

16.3.3. Technical Background Document and Response to Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April 1989

- 16.3.4. SOP QA-003, Quality Control Program
- 16.3.5. SOP CORP-IP-0003NC: Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods
- 16.3.6. SOP NC-MT-012: Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis, Method 6010B and Method 200.7
- 16.3.7. SOP CORP-MT-0005NC: Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1
- 16.3.8. SOP CORP-MS-0002NC: Determination of Volatile Organics by GC/MS based on Methods 8260B
- 16.3.9. SOP CORP-MS-0001NC: GC/MS Analysis Based on Method 8270C
- 16.3.10. SOP NC-GC-038: Gas Chromatographic Analysis Based on Methods 8000B, 8021B, 8081A, 8082, 8151A, and 8015B
- 16.3.11. NC-OP-0031: Extraction Procedure for Chloriantaed Acid Herbicides based on Method 8151A
- 16.3.12. SOP NC-OP-032: Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, and 600 Series Methods
- 16.3.13. SOP NC-QA-0017: Standards and Reagents
- 16.3.14. SOP NC-WC-0010: pH Electrometric Method
- 16.3.15. SOP NC-QA-0014: Glassware Washing

17. MISCELLANEOUS

17.1. Modifications/Interpretations from Reference Methods

- 17.1.1. Section 11.2: Preliminary Evaluations. Section 7.1 of the source Method 1311 states that the sample aliquot used for the preliminary evaluation “...may not actually undergo TCLP extraction.” Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., <

100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples which have been subjected to the oven-drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.

- 17.1.2. Sections 11.3.7 and 11.4.21: Determination of Filtrate/Extraction Fluid Compatibility. Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed.
- 17.1.3. Section 9.2: TCLP Extraction Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions “...that have been conducted in an extraction vessel.” TestAmerica has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e., Bottle or ZHE) per leach fluid used.
- 17.1.4. Section 11.2.5.5.8.1: Determination of Appropriate Extraction Fluid. Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.
- 17.1.5. Section 9.4: QA/QC - Matrix Spikes. Section 8.2 of the source method states “A matrix spike shall be performed for each waste type...” and “A minimum of one matrix spike must be analyzed for each analytical batch.” Further, Section 8.2.3 of the source method also states “The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist.” The standard TestAmerica QAM is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS and MSD will be prepared for each TCLP leachate batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results have immediate bearing only on the specific sample spiked and not all samples in the batch.
- 17.1.6. Section 8.2.2 of the source method states that “In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level.” The method also states “If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration but may not be less than five times the method detection limit”. For several analytes, spiking at the regulatory level is inappropriate to the range of analysis afforded by the determinative methods. Due to the wide range in these levels, TestAmerica spikes at the levels specified in the determinative SOPs.

APPENDIX A

TABLES

Table 3 - Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

Contaminant	mg/L
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

APPENDIX B

FIGURES

Figures 1 and 2 - Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

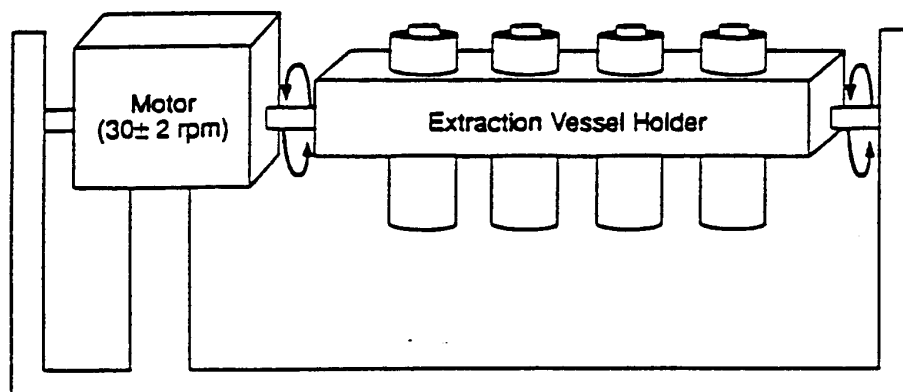


Figure 1. Rotary Agitation Apparatus

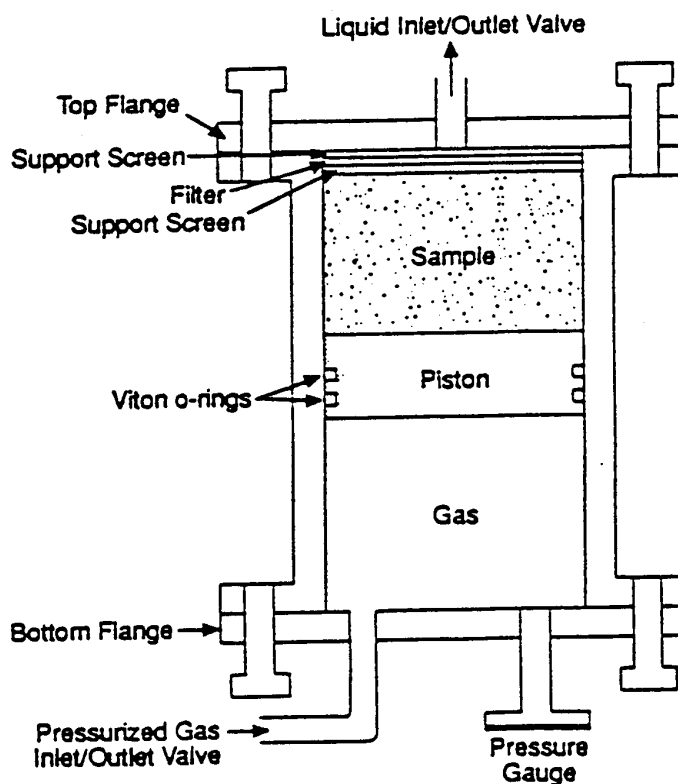


Figure 2 – Cross Section of Zero Headspace Extraction Vessel (ZHE)

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 1



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992
SUBJECT: Notes on RCRA Methods and QA Activities
From: Gail Hansen, Chief *Gail Hansen*
Methods Section (OS-331)

This memo addresses the following topics:

- 1992 Symposium on Waste Testing and Quality Assurance
- SW-846 Update
 - Final Rule for January 23, 1989 Proposed Rule
 - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- Environmental Monitoring Methods Index (EMMI)
- Sampling Work Group Formation
- MICE Update
- Oily Waste Analysis
- Electronic SW-846 Availability.

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 10 (cont'd)

Oily Waste Analysis

One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

APPENDIX C

LOGBOOK SHEETS

TESTAMERICA NORTH CANTON ZHE LOGBOOK

Pre- Prep Batch	Sample No.	Ext. Vessel ID	Matrix	Sample Wt (g)	Ext. Buffer ID	Date On	Time On	Analyst On	Date Off	Time Off	Initials Off	Tumbler ID	Tumbler Speed 30±2 RPM	Ext. Fluid ID	Initial ZHE Pressure	Final ZHE Pressure	Comments

TESTAMERICA NORTH CANTON ZHE LOGBOOK - MULTIPHASE

	A	B	C	D	E	F		G	H	
Sample Number	Weight of Container + Sample (g)	Weight of Container + Residue (g)	Difference (A – B) Sample Weight (g)	Weight of the Syringe (or vials) (g)	Weight of the Syringe (or vials) + Filtrate (g)	Difference (E – D) Filtrate Weight (g)	Volume of Filtrate* (mL)	Difference (C – F) Wet Solids Weight (g)	G x 20 Buffer Amount (mL)	Initials

* Filtrate volume only needed when filtrate is incompatible with leachate

TESTAMERICA NORTH CANTON TCLP LOGBOOK

Pre-Prep Batch	Sample No.	Parameters	Matrix	Pre-Test			% Dry Solids				Leach Start						Leach Finish				Tumbler ID	Comments
				Wt (g) Sample	pH after water	pH after heating	Wt (g) Sample	Wt (g) Filter	Filter + Particulates Wt (g)	% Dry Solids	Wt (g) Sample	Buffer # / ID	Date On	Time On	pH On	Analyst On	Date Off	Time Off	pH Off	Initials Off		

TESTAMERICA NORTH CANTON TCLP LOGBOOK – MULTIPHASE

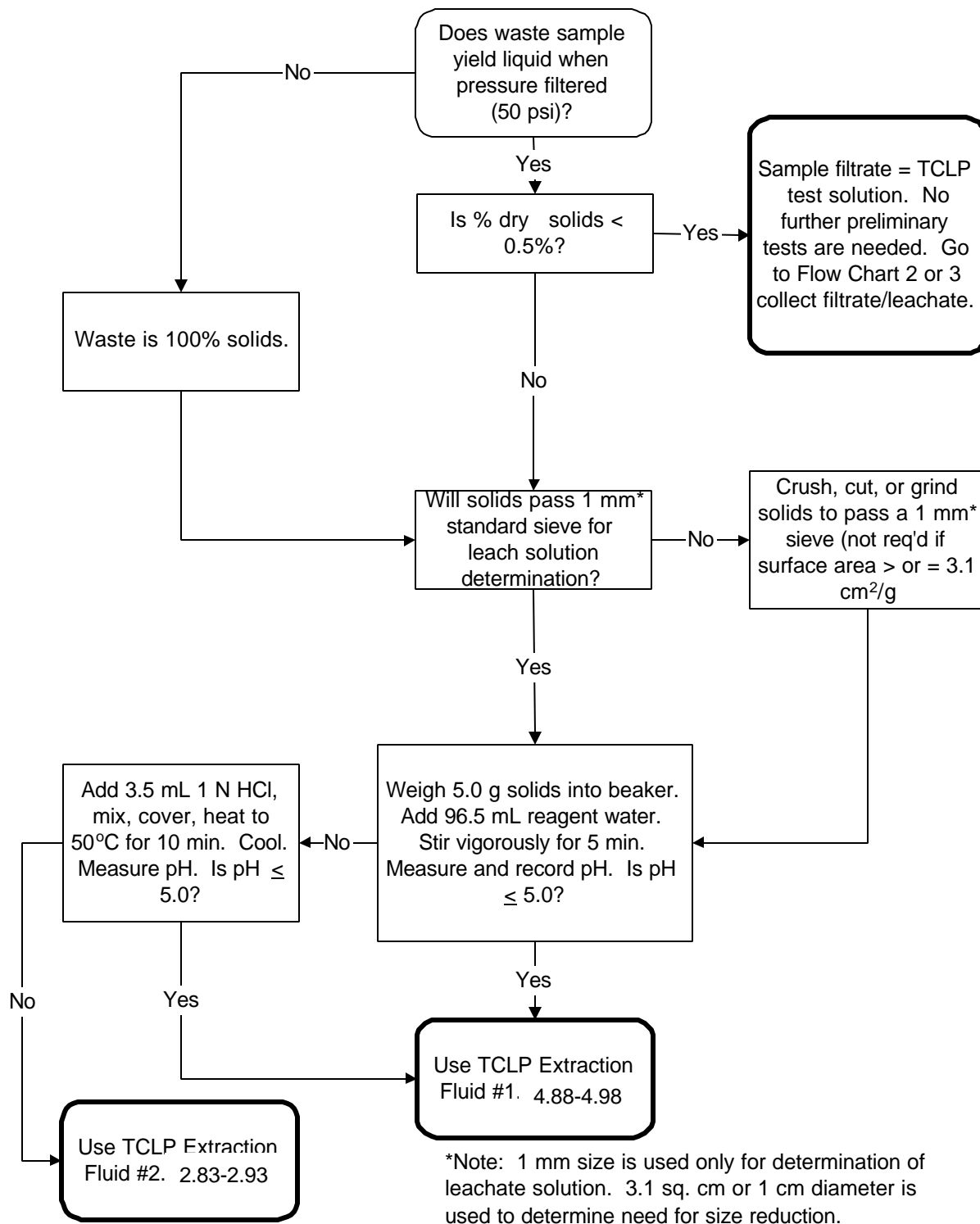
	A	B	C	D	E	F		G	H		
Sample Number	Weight of Container + Sample (g)	Weight of Container + Residue (g)	Difference (A – B) Sample Weight (g)	Weight of the Filtrate Bottle (g)	Weight of the Filtrate bottle + Filtrate (g)	Difference (E-D) Filtrate Weight (g)	Volume of Filtrate* (mL)	Difference (C-F) Wet Solids Weight (g)	G x 20 Buffer Amount (mL)	Initials	Comments

* Filtrate volume only needed when filtrate is incompatible with leachate

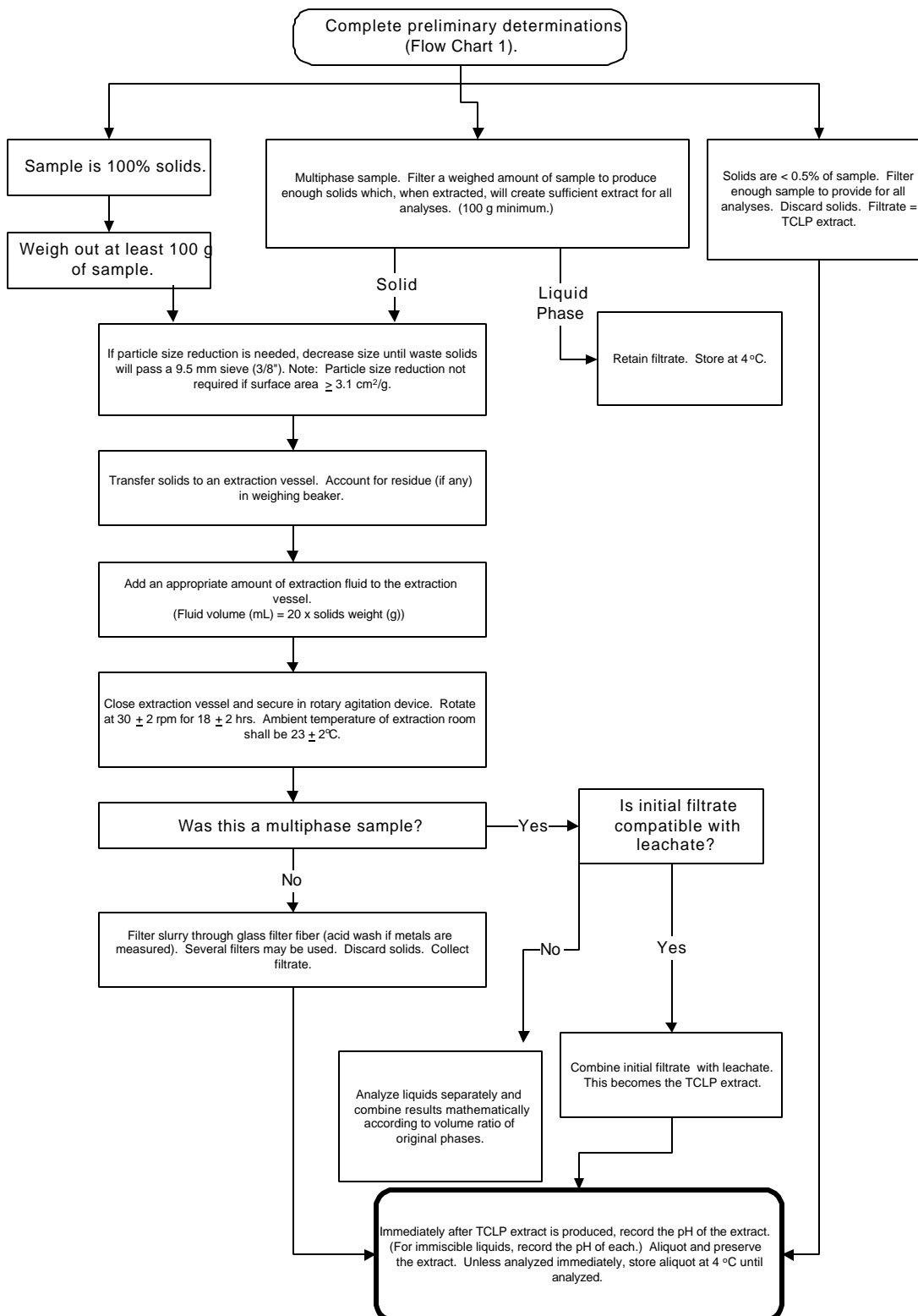
APPENDIX D

FLOW CHARTS

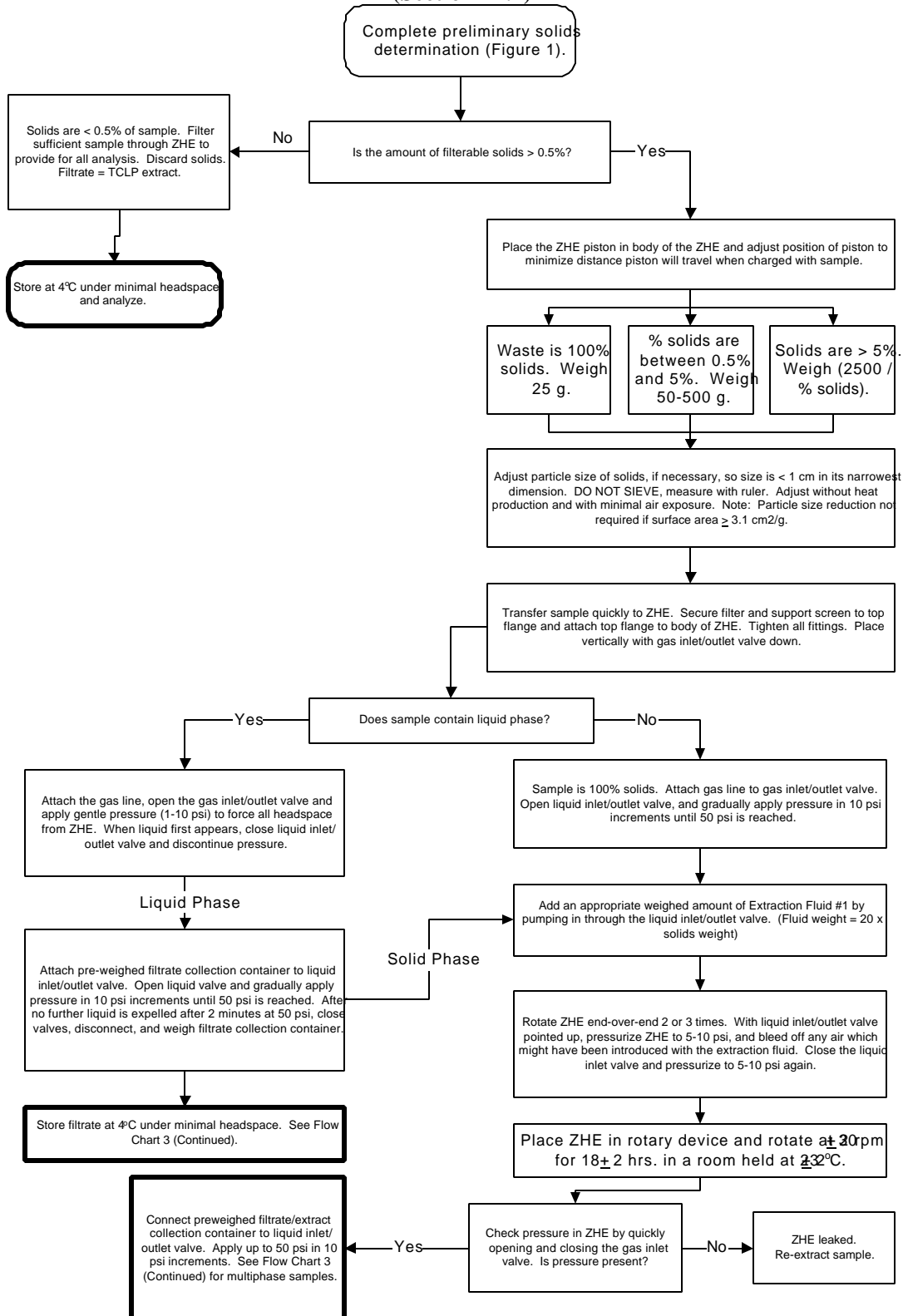
**Flow Chart 1. Preliminary Sample Evaluation
(Section 11.2)**



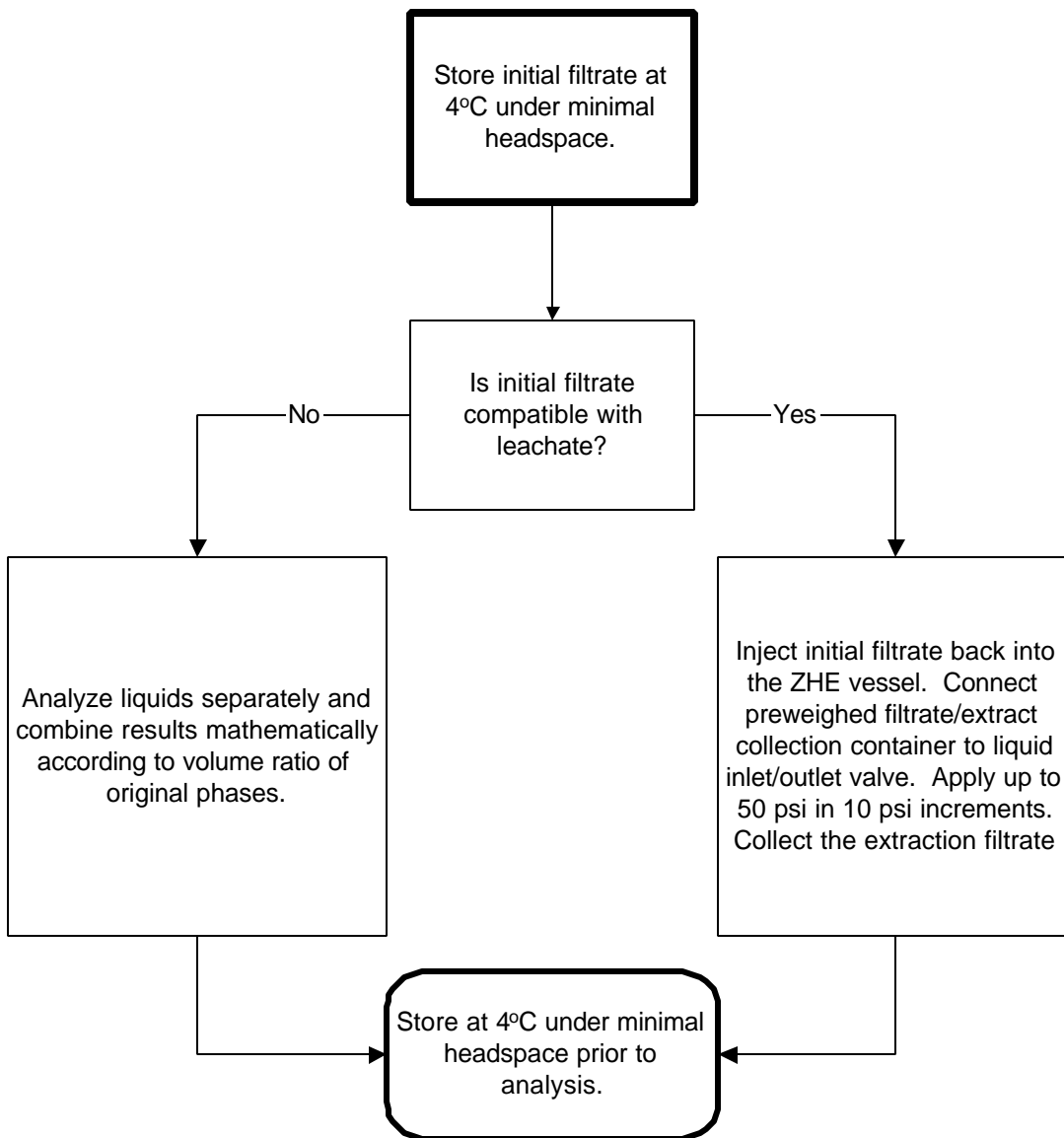
**Flow Chart 2. Bottle Extraction, Non-Volatile Constituents
(Section 11.3)**



**Flow Chart 3. ZHE Extraction, Volatile Constituents
(Section 11.4)**



**Flow Chart 3. ZHE Extraction
(Continued)**



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Implementation Date 9-27-07

SOP No. CORP-QA-0013NC

Revision No. 1.2

Revision Date: 09/18/07

Page 1 of 18

**TESTAMERICA NORTH CANTON
STANDARD OPERATING PROCEDURE**

TITLE: EMPLOYEE ORIENTATION AND TRAINING

(SUPERSEDES: Revision 1.1, 11/28/04)

Approved by:

Dorothy J. Reeson
Quality Assurance Manager

9/21/07
Date

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9/21/07
Date

Approved by

Mark Bruce
Technical Director

9/24/07
Date

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1. PURPOSE

- 1.1. To provide guidance in the establishment and maintenance of a formal documented training program for all TestAmerica North Canton laboratory and administrative support personnel. The training program is designed to provide a means for demonstrating competency, experience, and data quality and for stimulating professional development.

2. RESPONSIBILITIES

- 2.1. This SOP is applicable to all permanent or temporary technical and administrative support staff. Responsibilities of key staff members are as follows:
 - 2.1.1. *Associate* -- Each TestAmerica North Canton associate shall ensure that training received, as described in this SOP, is documented and that the documents are maintained in that associate's training file. Also, when formal external training is received, the associate shall forward pertinent records to the training files in the QA Dept.
 - 2.1.2. *Laboratory Director* -- The Laboratory Director is responsible for ensuring that the training requirements are implemented as delineated in this SOP. The Laboratory Manager is also responsible for ensuring that sufficient time is allocated to laboratory training and making final decisions on the qualifications of any trainee, if questions arise.
 - 2.1.3. *Quality Assurance Manager (QA) (or designee)* -- The QA Manager or designee is responsible for conducting orientation and on-going training in QA/QC policies and practices as described in this SOP. It is also the responsibility of the QA Manager or designee to maintain the documentation of training performed under this SOP at each TestAmerica North Canton facility.
 - 2.1.4. *Environmental Health & Safety (EH&S) Coordinator* -- The EH&S Coordinator or their designee is responsible for training new or transferred associates in accordance with the requirements of this SOP and with the TestAmerica North Canton EH&S Procedures. The EH&S Coordinator or their designee will ensure that necessary documentation is maintained and accessible for review by auditors. Documents will be archived according to TestAmerica North Canton filing procedures or electronically.

- 2.1.5. *Laboratory Operations Manager* -- Determines qualification required for technical positions and evaluates job candidates against those requirements. Certifies the qualification of laboratory personnel.
- 2.1.6. *Group Leader* -- The Group Leader is responsible for ensuring that all new and existing analysts and support staff in their group receive adequate on-the-job training and complete the initial and on-going proficiency demonstration requirements specified in this SOP. In addition, these individuals are responsible for continuously reviewing the analysts' performance and determining if additional training is required. This can be accomplished through the on-going peer or secondary technical reviews.
- 2.1.7. *Trainer* -- The trainer is responsible for providing the trainees with the instructions necessary to perform a given task in accordance with the appropriate SOPs and regulations. The trainer is also responsible for notifying the appropriate parties of the training that is to be conducted or has been conducted, as appropriate. The Trainer must forward all training documents to associate's training files. If required, QA or EH&S shall ensure the trainer meets the requirements for teaching the class. The trainer may be a staff member or from an outside resource.

3. SAFETY

- 3.1. Typical office-dependent safety precautions must be adhered to in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

4. PROCEDURE

4.1. Overview

- 4.1.1. Orientation and training are provided to achieve initial proficiency, maintain proficiency, and to promote continuous improvement. In general, new and transferred personnel are oriented and trained in the following subjects as they relate to his/her job requirements:

- TestAmerica North Canton EH&S Procedures and Corporate EH&S procedures
- TestAmerica North Canton QA Program
- Comprehensive Ethics Training
- Job-Specific Technical Procedures

4.2. Determination of Qualifications

- 4.2.1. TestAmerica North Canton shall identify basic job descriptions for all laboratory positions. These job descriptions shall provide the minimum qualifications for education and experience, knowledge and skills that are necessary for an associate to perform at a satisfactory level.
 - 4.2.1.1. A hard copy or electronic version of the current TestAmerica North Canton job description shall be included in the associate's training file or with Human Resources.
 - 4.2.1.2. An associate's performance shall be compared with the requirements of his/her job description annually as part of the associate's annual performance review that is conducted with the associate's supervisor.
- 4.2.2. A copy of the associate's resume may be included in the associate's training file or maintained in electronic format. The associate is responsible for ensuring the accuracy of the following information in the resume: academic credentials, employment history and experience and professional registrations.
- 4.3. Orientation to Environmental Health & Safety and Radiation Safety
 - 4.3.1. In accordance with the Corporate Safety Manual (CSM), it is TestAmerica North Canton policy to fully instruct all personnel who are exposed or potentially exposed to hazardous substances in basic safety operations prior to their initial work assignments.
 - 4.3.2. Each newly hired associate shall receive an EH&S Orientation as applicable to their job assignments to be conducted by the EH&S Coordinator or designee prior to being exposed to hazardous material or samples. The training will include the areas listed on the TestAmerica New Employee Training Checklist, CW-E-WI-004, Appendix D.
 - 4.3.3. Upon completion of the orientation, the associate shall take a follow-up exam 30-60 days after hire prepared by the EH&S Coordinator or designee as an indication that the contents of the training program is understood.
 - 4.3.4. Upon completion of the training, the EH&S Coordinator or designee shall submit the required safety training documents to the training files.
- 4.4. Orientation to Quality Assurance Program Requirements
 - 4.4.1. Each newly hired associate shall receive Quality Assurance Orientation Training and Comprehensive Ethics Training.

4.4.2. The Quality Orientation shall be conducted by the QA Manager or designee within the first 30 days of the laboratory associate's report-to-work date.

4.4.3. The QA Manager or designee shall provide orientation covering the following QA-related items as they apply to associate's assigned responsibilities:

- TestAmerica North Canton Quality Policy as described in the Laboratory Quality Manual (LQM), latest version
- TestAmerica North Canton's Ethics Policy on Data Integrity & Meeting Client/Contract Requirements
- TestAmerica North Canton Quality Documents
- Nonconformance & Corrective Action Identification and Documentation
- Proper Record Keeping Practices
- Key elements of TestAmerica North Canton QC Program Policy QA-003

4.4.4. In addition, the QA Manager or a designee shall provide other training in matters related to QA Program requirements on a routine basis. This will include the updates to the LQM or specific topics as related to the key TestAmerica North Canton QA Program elements. On-going QA training may be conducted in a meeting format, through publishing updated QA materials, or via presentations using computer media. Required QA on-going training shall be identified for each area within the laboratory.

4.4.5. The QA orientation or on-going QA training shall be documented using a written exam, a checklist, or a preprinted form. EXAMPLE forms that may be used for this purpose are illustrated in Appendix A.

4.5. On-the-Job Technical Training

4.5.1. On-the-job training shall be conducted by the Group Leader or an experienced peer immediately upon assignment of the laboratory associate to a given analytical preparation or determinative method.

4.5.2. The analyst shall become familiar with the procedure by reading the applicable Standard Operating Procedure (SOP). The trainee shall receive one-on-one instructions in performing the procedure from an experienced peer designated by management. The trainee shall work under supervision until the appropriate supervisor deems his/her work satisfactory.

4.5.3. On-the-job training documents shall be forwarded to the analyst's training files.

4.5.4. Authorization to work on client samples ***independently*** shall be granted when requirements specified in the following section are met. **Note:** Work may be performed by a trainee prior to the successful method proficiency completion only under the supervision of a qualified peer designated by management.

4.6. Demonstration of Analyst Proficiency

4.6.1. Initial Demonstration of Proficiency

4.6.1.1. An analyst must successfully prepare and/or analyze four consecutive laboratory control samples (LCSs) or a blind performance evaluation (PE) sample (if available) or if specified, meet EPA method requirements for number of proficiency samples. It is not necessary that each LCS be part of the same QC batch.

4.6.1.2. Each LCS must consist of a spiked aliquot of a control matrix (i.e., aqueous and/or solid, if available, and where there is a substantial difference in the methodology).

4.6.1.3. Control limits for accuracy and precision specified in the method SOPs or established by the laboratories will be used to assess the acceptability of data.

4.6.1.4. As appropriate, an associate must also demonstrate familiarity with proper documentation practices including but not limited to standards traceability, data review, nonconformance documentation, proper error correction protocols, and sample and document chain-of-custody procedures. Proficiency in performing these tasks can be accomplished by observation of the trainee by a supervisor, group leader, or a qualified peer.

4.6.1.5. If the results of QC samples do not meet the requirements specified in this SOP, the analysis must be repeated until the associate is able to independently demonstrate acceptable performance on another set of QC sample analyses.

4.6.2. On-going Demonstration of Proficiency and Retraining

4.6.2.1. The Quality Assurance Dept. is responsible for monitoring the associate's performance and evaluate his/her ability to perform adequately on each method. On-going proficiency can be demonstrated annually by:

(1) by the preparation and/or analysis of four consecutive LCS samples with acceptable results within laboratory-established control limits. This activity may be documented as part of the technical data review process; or

(2) a blind PE sample can be obtained from a regulatory authority, a client or generated internally. Examples of acceptable blind PE samples include WP/WS Studies, NELAC-approved PT studies, PEs submitted by the state agencies and the clients, double-blind PEs submitted by TestAmerica North Canton Corporate QA staff, or single blind PEs purchased from external vendors; or

(3) successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for 624 and 5035 [8260B]) would only require documentation for one of the test methods.

4.6.2.2.If internal (i.e., single blind) or external PE samples are used to establish proficiency, data must be compared against vendor's certified acceptance criteria or published study limits, respectively.

4.6.2.3.If the results of QC samples do not meet the requirements specified in this SOP, the analysis must be repeated until the associate is able to independently demonstrate acceptable performance on another set of QC sample analyses.

4.6.3. Documentation of Technical Proficiency

4.6.3.1.Acceptable performance shall be documented. The EXAMPLE forms provided in Appendix B can be used for this purpose. At any time an associate is no longer able to perform adequately, remedial training must be provided and proficiency process must be repeated and documented as described in Section 4.6.

4.7. External Training

4.7.1. In the interest of maximizing professional development and the long-term effectiveness of associates to meet projected needs, formal training in special fields may be provided through local institutions, training courses offered by the instrument vendors, scientific meetings, instructional workshops, and the U.S. Environmental Protection Agency (EPA).

4.7.2. The associate shall forward a copy of any certificate of training to the training files.

4.8. Requirements for Trainers

4.8.1. In all cases, training will be conducted by qualified staff selected on the basis of knowledge, experience, and proven competency. Technical, procedure-specific training may be conducted by experienced personnel familiar with the procedure, including the Laboratory Manager, Department Manager, Group Supervisors, Technical Specialist, and Associates who have either developed the procedure (SOP) or have already been qualified to perform the procedure. For administrative, health and safety, and QA-related procedures, the Department Manager, Health and Safety Manager, and QA Manager or a designee may conduct training as well as qualified associates who have either written the SOP or have already been qualified to perform the procedure. If required, the trainer shall take the necessary steps to meet the requirements for teaching the class.

4.9. Transfer of Analysts Between Facilities

4.9.1. An associate may be trained at one facility and transferred to another facility for temporary or permanent assignment. In the case of permanent assignment, the associate's training records must be transferred to the new location. As deemed necessary by the Management Group, the associate may need to further demonstrate acceptable performance as outlined in section 4.6 as part of the first set of samples analyzed at his/her new location.

4.9.2. The laboratory operations groups must maintain the analytical data generated during this process in the data archives.

4.9.3. A new Health and Safety orientation must be conducted with the employee per section 4.3 for the new facility.

4.10. Documentation for Technical Training Sessions

4.10.1. To demonstrate each associate's participation in a training course, the following minimum level of information must be included in the training files:

- The date of training,
- The name of trainer,
- The name of trainee (associate),

- The course/training session title which links to a reference set of training materials, and
- For technical training in analytical methods, attach qualifying data or include applicable references to the qualifying data such as a project number or analytical/QC batch to reference any analytical raw data substantiating successful training.

4.11. Maintenance of Training Records

4.11.1. At a minimum, a training file shall be maintained for each associate in an area designated by the Laboratory Director. Evidence or documentation of external training activities (if any) must also be kept for reference purposes. Training records must be maintained and managed under routine TestAmerica North Canton policies.

4.11.2. Each TestAmerica North Canton associate's training file shall include the following types of information:

- General (e.g., resume, job description, ethics related training)
- Health & Safety
- Quality Assurance
- Technical Proficiency Demonstration
- Professional Development (if applicable)

Note: All documentation for the above may be maintained in electronic files rather than as hard copy in individual's training file.

4.11.3. A database, although not required, can be used to record and monitor training for each associate and to notify management when retraining is necessary.

4.11.4. EH&S training files must be maintained in a such a manner as to be readily accessible on request (to auditors, clients, associates, or representatives) without being mixed with training records from other disciplines.

5. DEFINITIONS

5.1. *Orientation* -- The process by which an individual becomes acquainted with existing systems, environment, and conditions.

- 5.2. *On-the-job Training* -- A mechanism to help an associate to develop skills and introduction to local procedures.
- 5.3. *Technical Proficiency* -- A process by which analyst and method performance is demonstrated on the basis of generating acceptable QC data and consistent with TestAmerica North Canton QA policies.

6. REFERENCES

- 6.1. TestAmerica North Canton Laboratory Quality Manual (LQM), current version
- 6.2. TestAmerica North Canton Policy QA-003: TestAmerica North Canton Quality Control Program (current version).
- 6.3. U.S. EPA Interim Draft Requirements for Quality Management Plans (EPA-QA-R-2, July 92).
- 6.4. Current NELAC (National Environmental Accreditation Committee) Standards.

7. APPENDICES

Appendix A: *EXAMPLE* - Departmental Orientation Checklist

Appendix B: *EXAMPLE* - Technical Proficiency Documentation

Appendix C: *EXAMPLE* - Demonstration of Capability Certification Statement

Appendix D: *EXAMPLE* - New Employee Safety Training

APPENDIX A**EXAMPLE****TestAmerica North Canton
Departmental Orientation Checklist**

This document is due back to the HR Department within 30 days.

Employee's Name:	Department: _____
Trainer's Name:	Employee's Signature: _____
	Trainer's Signature: _____

	Trainee's Initials/Date	Trainer's Initials/Date
INTRODUCTIONS		
Key Personnel (Lab Director, Ops Mgr., Dept. Heads)		
Co-workers (tasks, length of time in dept, etc.)		
TOUR		
Instrument Area		
Data Area		
Prep Area		
SUPPLIES (CONSIGNMENT AREA)		
RELATIONSHIP OF DEPARTMENT TO TESTAMERICA, TO NORTH CANTON LAB		
Process/Flow of Samples through dept.		
Workflow from/with other departments		
Internal clients		

KEY RESOURCE INDICATORS (OTD, TAT, HTV)		
POSITION WITHIN THE DEPARTMENT		
Job Description		
Individual Responsibilities		
Maintenance/Housekeeping		
Backlog		
Communication		
REPORTING STRUCTURE (CHAIN OF COMMAND)		
Within the group/group leader		
Lab Structure		
Company Structure		
MISSION STATEMENT, VISION AND ETHICS		
Company Mission, Goals and Ethics		
Lab Goals		
Departmental Goals		
Following SOP		
Documentation of Anomalies (NCMs)		
Due Dates, HTVs		
DEPARTMENTAL POLICIES AND PROCEDURES		
Vacation, Holidays, Working Hours, Break Periods		
Timesheet		
Performance Review Schedule		
Job Expectations		
Benefits		
Ordering		
Consignment Area		
Conduct with Auditors		
	Trainee's Initials/Date	Trainer's Initials/Date
TELEPHONE AND COMPUTER SYSTEM		
Phone Training		
Target Account Training		
Internet/Intranet		
Email		

Clouseau		
AS400 Account Training		
Office Supplies		
INTERNAL WORK FLOW		
Paper flow for each sample lot		
Lot Summary		
Internal Work Flow (Cont.)		
Backlog		
Sample Worksheets		
Level I Review		
Level II Review		
NCM/Clouseau		
Batching		
HEALTH AND SAFETY		
Location & how to use Safety Showers, Eye Wash Stations		
Location of Emergency telephone numbers		
Emergency evacuation routes		
Meeting spot for fire evacuation, severe weather		
Reporting of Accidents/Exposures		
How to handle spills		
MSDSs (how to find on-line and location of backup disc)		
QUALITY ASSURANCE/QUALITY CONTROL		
Schedule Comprehensive Ethics Training with QA		
Read SOPs , including safety sections.		
Method specific training checklist are located at N:QAQC/training/checklists		

APPENDIX B

EXAMPLE

**TESTAMERICA NORTH CANTON
TECHNICAL PROFICIENCY DOCUMENTATION**

TYPE OF TRAINING: PERFORMANCE EVALUATION PROFICIENCY

I verify that _____ has been trained and met the technical proficiency requirements specified in the SOPs:

for the following *required* procedure(s):

Performance Evaluation Demonstration:

☐ Successfully prepared and/or analyzed an internal “blind” performance check sample.

(Reference: _____)

Performance Evaluation Demonstration:

☐ Successfully prepared and/or analyzed the following *external* performance evaluation sample.

Signatures:

Analyst: _____ Date: _____

Group Leader: _____ Date: _____

Quality Assurance _____ Date: _____

APPENDIX C**EXAMPLE*****Analyst Demonstration of Capability
Certification Statement*****Analyst Name****Date:***Test Method:**SOP:**Matrix: Water*

TestAmerica North Canton laboratory
4101 Shuffel Drive NW
North Canton, OH 44720
(330) 497-9396

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the TestAmerica Quality Assurance Plan, has met the Initial or Ongoing Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification following the TestAmerica SOP.
3. A copy of the laboratory-specific SOP is available for all personnel on-site.
4. The data associated with the initial/ongoing demonstration of capability are true, accurate, complete and self-explanatory (*). These data are attached to this certification statement.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/Observations:

Analyst's Name

Signature

Date

Technical Director's Name

Signature

Date

QA Manager's Name

Signature

Date

* *True: Consistent with supporting data.*

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices..

Complete: Includes the results of all supporting performance testing.

Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional




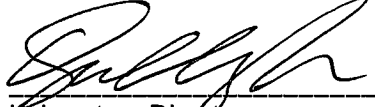
APPENDIX D**EXAMPLE**

NEW EMPLOYEE SAFETY ORIENTATION TRAINING CHECKLIST	
Employee's Name (Print)	Trainer's name (Print)
Date of Hire	Date of Training
The trainer must check each box as the training is conducted. The employee must initial the sections as indicated below. Indicate NA beside the check box where the subject is non-applicable for the employee.	
<p>_ Safety Manual Review _____ (Employee initials) I have read the most recent revision of the Corporate Safety Manual. I agree to abide by the policies and procedures in the manual. I understand that failure to do so could lead to disciplinary action up to and including termination of employment.</p>	
<p>_ Hazard Communication _____ (Employee initials) I understand that I must read the MSDSs for the material I use or someone must review the information in the MSDSs with me before I work with the materials. If an MSDS is not available, I will set the material aside until an MSDS can be obtained. _ Purpose of the Laboratory Standard (29 CFR 1910.1450) or Hazard Communication Standard (29 CFR 1910.1200) as appropriate. _ Location of MSDSs and how to obtain an MSDS. _ Location of additional safety information (i.e. Merck Index, SOPs, etc.)</p>	
<p>_ Emergency Procedures _____ (Employee initials) I understand that unless I have received training on the proper use of fire extinguishers, I am not authorized to use a fire extinguisher. If a fire occurs, I will immediately evacuate the work area. _ Location of emergency telephone numbers. _ Location of emergency assembly areas. _ Location and proper use of: _ Safety Showers, _ Eyewash Stations _ Fire Extinguishers _ Spill Equipment _ Exits _ Alarms _ Procedures to be followed if the following occur: _ A fire _ Chemical exposure _ Material spill _ Power outage _ Fume Hood fails _ Accident, Illness, or Near Miss _ Severe weather _ Workplace disturbance _ Earth quake _ Bomb/Terrorist Threats</p>	
<p>_ Personal Protective Equipment (PPE) Requirements</p>	

NEW EMPLOYEE SAFETY ORIENTATION TRAINING CHECKLIST	
Employees Name (Print)	Trainer's name (Print)
Date of Hire	Date of Training
<ul style="list-style-type: none"> – Eye Protection – Lab Coats – Gloves – Safety shoes or boots – Hard Hats – General Clothing – Use of respirators – Hearing Protection 	
<ul style="list-style-type: none"> – Waste Management Procedures <ul style="list-style-type: none"> – Discharge of materials to the sanitary sewer system – Typical wastes that are generated – Segregation of materials – Choosing an appropriate container – Preparation of the container – Labels – Accumulation Start Dates – Accumulation of the waste – Keeping containers closed – Head space – Use of secondary spill containers – Storage of waste in labs – Procedures to turn the waste in for disposal 	
<ul style="list-style-type: none"> – Transportation Safety <ul style="list-style-type: none"> – Shipments of DOT Hazardous Materials – Transportation Security Requirement – Personal use of company vehicles 	
<ul style="list-style-type: none"> – General Policies <ul style="list-style-type: none"> – Consumption of food, alcohol and tobacco products, and application of cosmetics – Proper use of fume hoods – Proper handling of glassware – Systems under pressure or vacuum/Transportation and use of compressed gas cylinders – Proper lifting of materials – Housekeeping – Working alone – Transition/Exclusion Areas – Access to medical records – Security and visitors in the lab 	
List any videos the employee watched as part of their orientation training:	
Employee's Signature:	
Trainer's Signature	

Title: RECORDS INFORMATION MANAGEMENT

[Method: None]

Approvals (Signature/Date):			
	4-11-08		4-11-08
Technology Specialist	Date	Health & Safety Coordinator	Date
	4/4/08		4/14/08
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-QA-019, Rev 6, dated 02/20/07

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Facility Distribution No. _____

Distributed To: _____

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1. PURPOSE

- 1.1. This document outlines the procedures associated with the storage and maintenance of all record created in the normal course of business such as laboratory data, client project files and all records generated in the course of laboratory operations.
- 1.2. This document accurately reflects current Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. RESPONSIBILITIES

- 2.1. It is the responsibility of the employee to perform the procedure described herein in full compliance with this SOP.
- 2.2. It is the responsibility of the Laboratory Director, QA Manager, and Department Supervisor of this facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.
- 2.3. Records are stored in a secure area at all times and can only be removed if charged out.

3. SAFETY

- 3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.
- 3.2. Only 1.2 cubic foot boxes may be used to store all paper records due to risk of injury from larger sized boxes. These boxes have been found to weigh approximately 40 pounds on average.
- 3.3. When the forklift is used, individuals will have forklift training under OSHA standards. All safety equipment, such as a safety harness, will be used when operating the forklift.

4. PROCEDURES

- 4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.
- 4.2. Labeling Folders and Filing

- 4.2.1. Labels are printed in Central Reporting
- 4.2.2. The receptionist will align the client labels at the top score mark on the side-tab manila folders.
- 4.2.3. Folders are then given centralized reporting to be filed and initiate report and chromatograms. All reports and chromatograms are filed together to form a "Project File".
- 4.3. Records Transfer Process
 - 4.3.1. Record transfer forms are received in Record Management from all departments of the lab: Accounting, QA, EH&S, lab groups, etc. The reports/lab data is placed in a box by year, random order retention period, and a Record Transfer form is created for each box.
 - 4.3.2. The box is assigned an accession number (Cintas bar-coded label) and assigned a box location number. The date the reports are received are also written on the outside of the box, along with the client code range, Lab ID range and location. The label is stapled to the Record Transfer Form and both the accession number and box location is written on the form, copied and returned to the Records liaison. Then at scheduled times, the boxes that have been given labels are picked up and shelved.
 - 4.3.3. At no time will the Records Manager check to see if the information listed on the Record Transfer form is correct. It is the responsibility of the Records liaison to ensure the information is correct on the Record Transfer form.
- 4.4. Data Entry
 - 4.4.1. Open up Report Tracker, click on "Menu", and click on "New Batch".
 - 4.4.2. The next batch number will automatically appear.
 - 4.4.3. In the Customer Number Box, click on the down arrow; and select the appropriate customer code as it appears on the Record Transfer form.
 - 4.4.4. Entry date will automatically default to the current date.
 - 4.4.5. Click on the arrow, and the next screen will appear for adding a new ascension number.
 - 4.4.6. Click on "Add New Ascension"

- 4.4.7. In the Ascension Number field, enter the Andrews bar code number that was assigned to the Transfer form. Click “OK”.
- 4.4.8. Click on the down arrow in the Record Series column. Select and click. Series will appear in the box.
- 4.4.9. Enter the box location that was assigned on the Record Transfer form. Example: 30-D-2A.
- 4.4.10. In the Document Description box, enter the information that is located on the Record Title of the Record Transfer for (no more than 50 characters).
- 4.4.11. In the “Date Range From” field, enter the earliest month as it appears on the Record Transfer form. Example: January would be entered as 01/01/05.
- 4.4.12. In the “Date Range To” field, enter the latest month and last day of that month as it appears on the Record Transfer form. Example: January would be entered as 01/31/05.
- 4.4.13. Click on the “Review Date” field, delete the current date, enter the last month and day as the “Date Range To” field, enter the year as it appears on “Destruction Date” on the Record Transfer form. Example: If the month “From and To” were January, and the Destruction Date was a five-year retention, in the Review Date field, it would appear as 01/31/05.
- 4.4.14. If there are no details to add in the “Detail” field, click the arrow to next field and click back again. This will save all data entry. If you need to add another ascension number, repeat steps starting from Section 4.5.5.
- 4.4.15. If there are details to enter (such as Client Code, Lab I.D., etc.), after entering in the “Add New Ascension” field is complete, click the arrow at the bottom of the field. This will take you to the Details field.
- 4.4.16. Enter: Client Code, Lab I.D., Title, Case Number, SDG, if applicable. As it appears on the Record Transfer form. Once this is complete, click on arrow back to the “Add New Ascension” field.
- 4.4.17. If another ascension number is to be added, return to Section 4.5.5 and repeat.
- 4.4.18. After all data entry is finished in that batch, click arrows left and select “Print Batch”.
- 4.4.19. Print the batch out, and make two copies of each Record Transfer form. The original and one copy go to the originator of the form. The second copy and the Print Batch sheets are for the Records/Warehouse Manager’s records. The

Records Manager will Level I Review his copy with the Print Batch sheet, and make corrections as needed. He/she will then sign the "Print Batch" sheet as follows: "Level I Review, the date of review, and his/her initials". It will then be forwarded for Level II Review.

4.5. Editing a Batch

4.5.1. Open the Report Tracker. Click on "Edit Batch".

4.5.2. Enter the Batch Number. Click "OK". Click on the arrow, then click on the ascension number you wish to edit. Make the changes that are needed, and click arrow right to save changes. If changes need done in the "Detail" field, click arrow, make changes, and click arrows back left to save the changes.

4.6. Searching in the Report Tracker

4.6.1. Open Report Tracker. Click "Search". Enter details in the correct field, then click "Search".

4.6.2. Details can only be entered in one field at a time, except in the "Client Code" and "Lab I.D." fields. Multiple I.D. numbers and client codes can be entered here.

4.6.3. Once complete, in the "Search" mode, click "Back". This will return you back to the "Search" field.

4.6.4. Enter next item to search.

- Human Resource Records can be requested by: Human Resources and Directors
- Director Records can be requested by: Directors
- Medical Records can be requested by: Human Resources, Directors, and the Lab Nurse

4.7. Record Requests and Charge-Outs

4.7.1. There is a 30-day charge-out period for all records. The person who has charged out a record is responsible for that record until it is returned to the Records Department.

4.7.2. Records can be requested by stopping by, email, or calling the Records Department. A request for records must include the Lab ID, ascension number, or record title, including the year at a minimum. It is preferable to also include the client name and mail date.

- 4.7.3. Records that are stored off-site can be retrieved and delivered on Tuesday and Thursday. In an emergency, records can be retrieved from off-site storage within one hour at a cost of \$40.

4.8. Vital Records

4.8.1. SQL Server Backup Policy

- 4.8.1.1. The primary tool used for the backup of the SQL, Office and Target DB Servers will be Backup Exec for Windows NT (including the Agent for Microsoft SQL Server for NT). This will allow a singular backup format and strategy for the server's operating system, data files and databases.

- 4.8.1.2. The backups are scheduled as follows:

Scheduled Job	Job Type	Frequency	Retention Period	Storage Location
Monthly	Full (including OS, data, and databases) – SQL and Office servers only	First Monday of Month 1:00 AM	1 Year	On-site Fire Safe
Weekly	Full (including OS, data, and databases) – SQL and Office servers only	Every Monday 1:00 AM	4 Weeks	On-site Fire Safe
Weekly	Full (including OS, data, and databases) – Target DB servers only	Every Monday 1:00 AM	Indefinitely	On-site Fire Safe
Daily	Differential (OS and data) Full (including OS, data and databases) – Target DB only Full (including OS, data, and databases) – Target DB servers only	Tuesday-Friday 1:00 AM	1 Week	On-site Fire Safe

- 4.8.1.3. The backups should be configured as follows:

- 4.8.1.4. SQL, Office Servers and Target DB

- 4.8.1.5. Server Backup Media Set Should have Overwrite Period = 7 day, Append Period = Infinite
- 4.8.1.6. Job Properties
 - 4.8.1.6.1. Append to media; overwrite if no appendable media is available.
 - 4.8.1.6.2. Device – Quantum 0
 - 4.8.1.6.3. Media Set – Server Backups
 - 4.8.1.6.4. Write Checksums to Media (True)
 - 4.8.1.6.5. Compression – Hardware (if available, otherwise software)
 - 4.8.1.6.6. SQL – DATABASE – Backup Entire Database
- 4.8.1.7. Backup Method for Files
 - 4.8.1.7.1. Full Backup – NORMAL – Back Up Files – Reset Archive Bit
 - 4.8.1.7.2. Differential Backups – DIFFERENTIAL – Change Files
- 4.8.1.8. Backup Selections
 - 4.8.1.8.1. Include all data directories
 - 4.8.1.8.2. SQL Server Databases include only non-QuantIMS objects of DATAMIRROR and include all other databases except PUBS and NORTHWIND.
- 4.8.1.9. The monthly backup tape consists of the first Monday of the month of the weekly backup tapes. There is no additional monthly backup performed.
- 4.8.1.10. All backups should be monitored and verified by the LAN Administrator. Execution of the backups, and tape handling is also the lab LAN Administrator's responsibility.
- 4.8.1.11. Once the tapes are brought to Records, they are logged in on an SQL Backup Tapes logsheet, and then returned to be recycled.

4.8.2. Blueprints

- 4.8.2.1. The building blueprints have been duplicated on aperture cards, which are stored off-site, in a vault at Cintas Records Management. A copy is kept on-site in a fireproof cabinet.

4.9. Record Retention

- 4.9.1. Records are retained for a total of five years unless a client specifies other retention requirements. Drinking water analyses are kept ten years, and NELAP requires five years
- 4.9.2. In the event that the laboratory transfers ownership or goes out of business, the records will be maintained until the scheduled retention period has been met. Clients with contractual agreements for the return of their records will be contacted. Those records will be returned at the client's expense. Proof of ownership and responsibility will be required before the records may be released.
- 4.9.3. This procedure ensures compliance with the TestAmerica Record Retention Policy, P-L-001.

4.9.3.1. Special Record Retention Requirements

Program	Retention Requirement
Ohio – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
OSHA - 40 CFR Part 1910	30 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement and others as negotiated.
Ohio Voluntary Action Program	10 years

- 4.9.4. At the end of the specified retention period, records are destroyed per criteria in SOP NC-QA-013, Inventory/Warehouse Control.

5. DEFINITIONS

- 5.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.
- 5.2. Chromatogram(s) refers to raw data generated from laboratory organic analyses.
- 5.3. Field - one part of record that contains specific information. (Example: the box number would be one field)
- 5.4. Record - consists of a number of information fields. (Example: the box number, location and department would make up a record)
- 5.5. Batch - one or more boxes entered for a single department.
- 5.6. Accession number - six digit bar code number used to identify a single box.
- 5.7. File boxes - the standard box used is 1.2 cubic feet in size and weighs approximately 35 - 40 pounds full.
- 5.8. Vital records - records that are essential to protecting the assets of the company and its ability to continue operations are considered vital.
- 5.9. References
 - 5.9.1. CW-Q-S-001, Corporate Document Control and Archiving, current version
 - 5.9.2. P-L-001, Record Retention, current version
 - 5.9.3. NC-QA-013, Inventory/Warehouse Control, current version

6. APPENDICES

- 6.1. Example Records Transfer Form
- 6.2. Department Codes for Report Tracker

Appendix 1: Example Records Transfer Form

RECORD TRANSFER FORM

Directions: 1) Complete one form per box and detail contents as much as practical. 2) Write record title on side of the box with a handle and year of content. 3) Send Record Transfer Form approved and signed by Department Record Liaison to records. 5) After receiving barcode label attached under the box handle. 6) The location, which is to be written on the right side of box, (same side with handle) and circled. 7) Place Record Transfer Form in box. 8) Place box in centralized record pick-up area.

NAME:

DATE: (RELEASED BY)

Record Series Number: 0069 – QUAL ASSUR

Department Code: 00QA – Quality Assurance

Record Title:

(No more than 50 Characters) (Example: 2000 Sample Removal Requests, January 2000 Batch QC, etc.)

Date of Records:

Retention Period:

Destruction Date:

(One year per box ONLY)

(Refer to Retention Policy, LEG-004)

(Year Only)

Matrix: Paper

Film/Fiche

Diskette

Mag Tape

CD

Other

Record Liaison:

Accession Number:

Box Location:

Records Use Only:

Batch#: _____ **AS400 entry:** _____ **Report Tracker entry:** _____

Contents: (Be very specific. List when applicable: client code, lot number, name of client, batch numbers, instrument number, month date range, etc.)

N:\QAQC\SOPS\QUALITY\SOPs in Revision\NCQA019 Records Rev 7.doc

Rev 2, 02/22/07 jse

Appendix 2: Department Codes for Report Tracker

Code	Department	Code	Department
00HR	Human Resources	0MSS	GC/MS Semivolatiles
00IS	Information Services/Technology	0MSV	GC/MS Volatiles
00PM	Project Management	0WET	Wet Chemistry
00QA	Quality Assurance	MAIN	Maintenance
0ADM	Administration	OPER	Operations
0EHS	Environmental Health & Safety	PURC	Purchasing
0EXT	Organic Extractions	RMGT	Records Management
0FAS	Field Analytical Services	SALE	Sales
0GCS	GC Semivolatiles	SAMC	Sample Control
0GCV	GC Volatiles	SHIP	Shipping
0LAW	Legal	TRAI	Training
0MET	Metals		

Controlled Copy

Copy No. _____

Implementation Date: 7-25-07

SOP No.: NC-QA-0027

Revision No.: 1.2

Revision Date: 07/15/07

Page 1 of 16

TestAmerica North Canton
STANDARD OPERATING PROCEDURE

TITLE: Preparation and Management of Standard Operating Procedures

(SUPERSEDES: Revision 1.1, Revision Date 12/10/04)

Approved By: Wendy J. Leson 7/19/07
Quality Assurance Manager Date

Approved By: Paul G. H. 7/23/07
Laboratory Director Date

Approved By: Mark T. Bune 7/19/07
Technical Director Date

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1. Purpose

- 1.1. To identify the requirements for the development, review, and management of facility standard operating procedures (SOPs) and the requirements for controlled distribution of SOPs.

2. Responsibilities

- 2.1. The QA staff shall be responsible for the document control system for SOPs, including maintenance of the original documents and historical files, controlled distribution of new or revised SOPs, documentation of circulation, and issuance of unique, sequential SOP numbers. The QA staff is also responsible for replacing superseded SOPs with the new revisions.
- 2.2. Operations Management, the Customer Service Manager, the Technical Director and the QA Manager shall be responsible for writing and reviewing the SOPs specific to their areas. They are also responsible for the implementation and the strict adherence to SOPs.
- 2.3. Each associate shall be responsible for following all SOPs pertaining to activities in his/her work area.

3. Safety

- 3.1. Normal office-dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.
- 3.2. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to the EH&S Coordinator and a laboratory supervisor.

4. Procedure

- 4.1. All routine management, administrative, and operational activities associated with the production of analytical data at TestAmerica North Canton must be documented using SOPs or supported by written policies. The format of the SOPs shall be determined based on the type of procedure being described. The required formats are provided in the appendix of this procedure.

- 4.2. SOPs must be developed, reviewed, approved, implemented, distributed, and maintained using the formats and procedures described in this procedure.
- 4.3. SOPs shall be generated using an appropriate word-processing software in standard, consistent font and type sizes. All sections shall be numbered according to the outlines provided in the appendices of this policy. Subparagraphs shall be numbered using standard decimal numbering practices (e.g., 1.1, 1.1.1, etc.) as applicable. Each paragraph should be single-spaced with a blank line between paragraphs. The document shall be typed in single column format.
- 4.4. The types of SOPs utilized in the laboratory operations may include corporate SOPs and laboratory SOPs. In developing SOPs, the author must review the SOP for consistency with requirements specified in TestAmerica's Quality Management Plan (QMP), the Laboratory Quality Manual (LQM), Corporate Safety Manual (CSM), policies, method or regulatory requirements and existing SOPs.
- 4.5. If clients require the use of a procedure which deviates from an existing SOP, the requirement and use of the alternate SOP must be specified in the project documents (e.g., QAPjP, NCM, etc.) or in contractual documents. The title, number, and contents of this SOP must clearly indicate that the procedure is developed to meet a specific project's requirements. Client specific addendums and sections of SOPs can be added as long as they clearly indicate that the procedure is specific to a particular project or client.
- 4.6. SOPs are numbered using the numbering system described in Appendix I. Each page must include the header with the SOP number, the Revision Number, the Revision Date and the page number, including total number of pages (right justified) and the SOP Title (left justified), as shown in Appendix II. If the document supersedes a previous revision or replaces a different SOP, this is listed on the title page. If no document is superseded, state "None". The revision number indicates the number of times the document has been revised. Each revision will necessitate a new Revision Date. The revision number is designated with an integer 1,2,3, etc. Draft Documents should be labeled as Draft Revisions. The first issuance of a document is Revision 1. Approval of operation SOPs is indicated by signature and date of the appropriate personnel on the cover sheet of the SOP. The header information and signature requirements are shown in Appendix II. SOPs shall not be used to perform work unless they are controlled copies of the approved document, indicated by the attached and completed signature page. Uncontrolled copies of SOPs can be distributed to Operations management for review before the procedure is implemented or as a personnel copy for the analyst. It is distributed and printed via the laboratory's intranet. All such print outs contain a

watermark outlining that it is an intranet controlled SOP and has a print date on the cover page.

- 4.7. The effective date of the SOP is the laboratory designated implementation date, the date when controlled copies are distributed and the document is first put into use at TestAmerica North Canton. The implementation date is indicated on the title page as such. The implementation date is to be filled in by QA at the time of controlled document distribution. The document control systems must include a master list or file that identifies the current revision status of quality documents and records the effective date for each update and version of the documents so that the history of use for each at the facility can be demonstrated.
- 4.8. SOPs are proprietary of TestAmerica North Canton and are confidential documents. The proprietary information statement displayed on the signature page of the SOP (see Appendix II) is an integral part of the document and must be affixed to each approval page of SOPs. TestAmerica North Canton SOPs are proprietary documents; therefore, they should not be routinely disseminated to clients or other external entities without the permission of the responsible lab manager or QA manager.
- 4.9. Work Instructions can be used to document simple procedures and/or project-specific requirements as placards in labs. These documents are not required to undergo the same level of control as official SOPs. However, Work Instructions must be approved by a QA Department representative, have the appropriate SOP reference, revision number, and date.
- 4.10. SOPs are normally controlled documents. They shall be distributed for use internally as “controlled” documents and externally as “uncontrolled” copies, unless requested as such by the client. Only copies of the complete SOP, including approval signatures, shall be distributed. For example, Department of Energy (DOE) requires controlled copies of SOPs and other laboratory documents.
- 4.11. Controlled copies are numbered and their distribution documented using an SOP Receipt Form (see example in Appendix III). This form may be computerized and the records maintained in a database. All personnel receiving controlled copies of SOPs must document that they have received the SOP(s) using a SOP Circulation List. The SOP Receipt Form (see example in Appendix III) may also be used to document SOP receipt and is placed in the appropriate training files.
- 4.12. SOPs sent to clients or government agencies must be issued by the QA Department as uncontrolled copies, unless specific requirements to provide controlled copies exist. The recipients will need to be tracked only if the documents are controlled. The SOP

number, revision number, along with the client name and address and contact person, must be tracked for controlled copies. The uncontrolled copy must be marked "UNCONTROLLED COPY" in the area of the Controlled Copy Number blank on the signature page. Uncontrolled copies are not numbered.

- 4.13. The current, approved SOP must be followed each time the covered activity is performed, unless a contract or QAPjP requires a specific version of a SOP. Client agreements that reference specific SOPs should be written where feasible, to incorporate subsequent revisions of these SOPs.
- 4.14. Following the completion of a SOP, each SOP must undergo the required review process before final publication. Each SOP must be reviewed by a representative staff member of the following sections: (1) the author, (2) appropriate section supervisor, manager, technical lead or qualified peer, (3) QA Department, (4) Environmental, Health and Safety, and (5) the Lab Manager.
- 4.15. Format, Development, and Approval of Standard Operating Procedures:
 - 4.15.1. Corporate SOPs are documents developed by corporate personnel for use across the company where standardization of procedures is deemed necessary. The format for Corporate SOPs is described in TestAmerica Policy S-Q-001, current revision, "Official Document Control and Archive."
 - 4.15.2. The format for laboratory SOPs are described in Work Instructions listed in Reference Section 6.8 The table in Appendix IV provides the cross references between the TestAmerica QMP, TestAmerica North Canton SOPs, LQM and other documents that address the laboratory methods manual (SOPs) contents requirements specified in Section 5.10.1.2 of the NELAC Quality Systems Standard, current version.
 - 4.15.3. Laboratory SOPs shall be developed for all routine procedures that are not described in corporate SOPs. Final approval of laboratory SOPs shall be designated by signatures on the cover page as specified in section 4.14
- 4.16. Document Control for Standard Operating Procedures
 - 4.16.1. Distribution
 - 4.16.1.1. SOPs shall be written and approved according to the instructions described in this procedure. Once the SOP has been approved, it must be implemented as written. All individuals required to perform the

procedures described by a SOP must read the document and undergo training under the direction of the section supervisor or group leader. For new procedures or major changes to existing procedures, adequate training in addition to reading the SOP must be provided by the section supervisors or group leaders to ensure proper implementation. Such training shall be documented in the associate's training file.

- 4.16.1.2. Controlled distribution and maintenance of the SOPs is the responsibility of the QA Office. The SOP files consist of the original document file and satellite files. The original document file contains copies of all SOPs issued by corporate management and original SOP documents issued by the facility. Satellite files, if used, contain photocopies or electronic copies of any SOPs distributed to work areas as may be required. Electronic copies are kept on the public drive where security of the documents are maintained, and they are available to the user. Controlled centralized hard copies are also available.
- 4.16.1.3. When the SOP is revised, the previous version should be removed unless it is still in use. The satellite files may contain all, or an appropriate subset of the corporate and laboratory SOPs as they apply to a specific function. Distribution of SOPs to associates and/or all satellite files shall be documented. An SOP Receipt Form provided in Appendix III is an example of the type of required documentation. Distribution records may be computerized. All SOPs shall be marked as controlled or uncontrolled copies as described above. Any associate issued one or more personal copies of SOPs shall document the receipt of the SOPs on a SOP Receipt Form (Appendix III). This policy allows for use of electronic forms and electronic distribution of controlled and uncontrolled copies. Electronic receipt can be done via electronic mail (email) using receipt acknowledgment.

4.17. Revision

- 4.17.1. SOPs must be revised if there is a substantial change in the procedure. In the case of planned analytical methods modifications, an approved revision of the SOP should be in place prior to implementation of the modified procedure in the laboratory. Minor SOP revisions may be documented through the use of the SOP Change Form (see Appendix V) which also must undergo the same review process as the SOP and/or Non-conformance memo (NCM). One-time changes to the SOPs due to problems associated with instrument, matrix,

etc. may be noted also via NCMs. These minor changes must be incorporated into the body of the SOP when the SOP is revised.

- 4.17.2. For laboratory SOPs, the completed change form must be approved by the technical supervisor, the QA Manager, the Environmental Health & Safety Coordinator, and the Lab Manager, until edited copies are completed. Revisions to corporate SOPs can only be made by the same approval process used for the corporate SOP as indicated by the same signature authorities of the approved SOP.
- 4.17.3. Handwritten changes without the approval of the Section Supervisors or Manager and Quality Assurance department in SOPs are prohibited. Handwritten changes are considered “interim” and are only allowed if each modification or addition is accompanied by the initials and date of the Operations Management and QA using the SOP Change Form. The SOP Change Form can be used to document interim, minor revisions. This form must be approved, including the appropriate signatures, same as the SOP.
- 4.17.4. It is TestAmerica policy to review each SOP every two years. If during the every two-year review, it is determined that no modifications are necessary, an SOP Review Form (Appendix VI) can be used to extend the life of the document until significant changes occur. This form must be reviewed by the reviewer (a representative of a department) and QA. During this process, the SOP may or may not need to be reissued with a new revision date and number. The SOP revision is updated in the SOP database and the Review Form is maintained in the QA files. There are some programs that require review at different time frames. Refer to specific project set-up requirements (e.g. Supplemental Practices for DoD Project Work SOP, NC-QA-0016)
- 4.17.5. Each revision must be numbered in sequential ascending order. When the revised SOP is distributed to the laboratory, all copies except those required for projects must be removed from use by the QA staff or designee. The original document of the preceding revision is placed in a historical file and maintained according to the requirements of the Record Retention Policy.

5. Definitions

- 5.1. Document control system: The system of document generation, review, approval and reproduction that will ensure the use of the most current, applicable procedures in all areas.

- 5.2. Controlled copy: Controlled documents distributed with the intention that all updates of that document are provided to the holder of the controlled. Each copy is marked “Controlled Copy”. The copies are numbered and a controlled copy distribution list (e.g., SOP Circulation List) is maintained which includes the holder’s name and document number.
- 5.3. Uncontrolled copy: A copy of a controlled document that is issued with no intent to provide the holder with subsequent revisions. Generally, uncontrolled copies are distributed to external clients. The document is marked “Uncontrolled Copy.” An uncontrolled copy is not numbered.

6. Appendices

- 6.1. Appendix I: SOP Numbering System.
- 6.2. Appendix II: Example Title Page for Facility SOPs (Laboratory SOP).
- 6.3. Appendix III: Example SOP Receipt Form.
- 6.4. Appendix IV: Table of Cross References Between the TestAmerica QMP, LQM and Method SOPs that Address NELAC Laboratory Method Manual Specifications (07/01/99).
- 6.5. Appendix V: Example SOP Change Form for laboratory SOPs.
- 6.6. Appendix VI: Example SOP Review Form.
- 6.7. References
- 6.7.1. WI-NC-009A_032907, SOP Non-lab Template, current version.
- 6.7.2. WI-NC-008A_032907, SOP Lab Template, current version

APPENDIX I: SOP NUMBERING SYSTEM

SOPs are numbered to identify the source and application of the document as follows:

Location-Function-####

where:

Location:

XXX = Your city

Function:

QA = Quality Assurance

GC = Gas Chromatography

LC = HPLC

MS = Mass Spectrometry

MT = Metals

WC = Wet Chem

IP = Inorganic Prep

OP = Organic Prep

IT = Information Technology

HS = Environmental Health and Safety

AD = Administrative

SC = Sample Collection

= Number assigned sequentially beginning with 0001.

Client-specific SOPs can be identified by a 2-4 letter abbreviation of the client name at the end of the numbering system (e.g. NC-MS-0001GM).

NOTE: Facility versions of former Quanterra corporate SOPs may be numbered as CORP-Function-XXX and lab location abbreviation at the end. This numbering system will be phased out as these SOPs are revised.

APPENDIX II: Example Title Page for Facility SOPs (Laboratory SOP).

Controlled Copy

Copy No. _____

Implementation Date: _____

SOP No. _____

Revision No. _____

Revision Date _____

Page 1 of _____

**TESTAMERICA NORTH CANTON
STANDARD OPERATING PROCEDURE**

TITLE: _____

(SUPERSEDES: _____)

Prepared by: (Enter Name of Preparer or Reviewer) _____
Date _____

Approved by: _____
Technical Specialist Date _____

Approved by: _____
Quality Assurance Manager Date _____

Approved by: _____
Environmental, Health and Safety Coordinator Date _____

Approved by: _____
Laboratory Manager Date _____

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APPENDIX III: EXAMPLE STANDARD OPERATING PROCEDURE RECEIPT FORM

**STANDARD OPERATING PROCEDURE
RECEIPT**

I have been issued the listed SOP/Policy. I have read and understand that I must comply with the instructions of this SOP/Policy to perform my work for this process. I shall document any deviations which occur from this SOP/Policy and bring them to the attention of my supervisor. I understand that, at any time, I can bring to the attention of my supervisor or the QA Department any questions which I have during the performance of my duties according to this SOP/Policy.

The latest version of this SOP/Policy is available electronically in read only format at N\QAQC\SOP\ (lab group)\ (SOP).

SOP NO.	Title	Revision No.	Revision Date	Control Copy No.

NAME	SIGNATURE	DATE

Please return to QA after you have signed and dated. Thank you!!
THE CHANGE FORM IS ATTACHED SO YOU CAN EASILY SEE THE
CURRENT CHANGES MADE.

Appendix IV: Table of Cross References Between the TestAmerica QMP, LQM and Method SOPs that Address NELAC Laboratory Method Manual Specifications

NELAC 5.10.1.2 Laboratory Methods Manual	TestAmerica NORTH CANTON Method SOPs	Supplemental Sources Common to All SOPs
1. Identification of the test method	Scope and Application	QIMS SACs
2. Applicable matrix or matrices	Scope and Application	QIMS SACs
3. Detection limit	Scope and Application	QIMS SACs
4. Scope and application, including components to be analyzed	Scope and Application	QIMS SACs
5. Summary of the test method	Summary of Method	Referenced Test Method
6. Definitions	Definitions	QMP 3.0 and LQM Glossary
7. Interferences	Interferences	Referenced Test Method
8. Safety	Safety	CHP
9. Equipment and supplies	Equipment and Supplies	LQM Table 8.0-1, Instrument List
10. Reagents and standards	Reagents and Standards	QMP 4.6 and LQM 5.2 Procurement of Supplies and Services
11. Sample collection, preservation, shipment and storage	Sample Collection, Preservation and Storage	LQM 8.5.2 Sample Containers, Shipping Containers, Preservatives, and Holding Times
12. Quality control	Quality Control	LQM 8.4 Quality Control Samples
13. Calibration and standardization	Calibration and Standardization	LQM 8.5.4 Calibration Procedures and Criteria
14. Procedure	Procedure	LQM 8.2 Analytical Methods
15. Calculations	Data Analysis and Calculations	LQM 8.8.2 Data Verification
16. Method performance	Method Performance	LQM 8.3 Data Quality Objectives

Appendix IV Table of Cross References Between the TestAmerica QMP, LQM and Method SOPs that Address NELAC Laboratory Method Manual Specifications, continued

NELAC 5.10.1.2 Laboratory Methods Manual	TestAmerica NORTH CANTON Method SOPs	Supplemental Sources Common to All SOPs
17. Pollution prevention	Pollution Prevention	CHP 14.2.5
18. Data assessment and acceptance criteria for quality control measures	Data Analysis and Calculations	LQM 8.6.1 Data Quality Assessment & TraQAr Control Limits Software
19. Corrective actions for out-of-control measures	Quality Control; Calibration and Standardization	LQM 9.1, 9.3 Nonconformance, Corrective Actions & TraQAr Clouseau
20. Contingencies for handling out-of-control or unacceptable data	Quality Control; Calibration and Standardization	LQM 9.1, 9.3 Nonconformance, Corrective Actions & LQM 8.9.4 Reporting Analytical Results
21. Waste management	Waste Management	CHP 14.2.5 & Facility-specific Waste Handling SOPs
22. References	References	LQM 8.2 Analytical Methods
23. Any tables, diagrams, flowcharts and validation data	Miscellaneous (Tables, Appendices, etc.)	--

CHP: Chemical Hygiene Plan

QMP: TestAmerica Quality Management Plan

LQM: TestAmerica NORTH CANTON Laboratory Quality Manual

QIMS = Laboratory Information Management System

SAC: **Structured Analysis Code** defines the analytical test requirements for each sample in terms of type of matrix, analytical method, extraction method, and QC program (e.g., reporting limits, QC limits, etc.)

APPENDIX V: EXAMPLE SOP CHANGE FORM FOR LABORATORY-SPECIFIC SOPS**TestAmerica NORTH CANTON SOP CHANGE FORM**

SOP NUMBER:	
SOP TITLE:	
SOP SECTIONS AFFECTED BY CHANGE:	
REASON FOR ADDITION OR CHANGE:	
CHANGE EFFECTIVE FROM:	
CHANGE OR ADDITION (SPECIFY SECTION; USE ADDITIONAL SHEETS IF NECESSARY)	
SUBMITTED BY/DATE:	
*APPROVED BY:	
Technical Review Signature	Date
Environmental Health & Safety Signature	Date:
QA Manager Signature	Date:
Laboratory Director Signature	Date:



APPENDIX VI: EXAMPLE SOP REVIEW FORM**SOP REVIEW FORM**

SOP NUMBER:	
SOP TITLE:	
<p>The subject SOP has undergone the required peer/management review. No modifications are necessary at this point. The SOP will be reviewed two years from the date indicated below.</p>	
REVIEWED BY/DATE:	
*APPROVED BY:	
_____	_____
Technical Review Signature	Date
_____	_____
Environmental Health and Safety Signature	Date
_____	_____
QA Manager Signature	Date
_____	_____
Management Signature	Date

*Must be same signature authorities of SOP being revised.

Title: DATA VALIDATION RESPONSE

Approvals (Signature/Date):

	<u>07/08/08</u>		<u>7/8/08</u>
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as Policy No. QA-020, Rev 3, dated 08/03/07

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OBJECTIVE

This policy describes the TestAmerica North Canton program of responding to client data validation inquiries. Data validation inquiries are received by the laboratory Project Manager for resolution. It is the responsibility of the Project Manager to be the central point of contact between the client and laboratory. The Laboratory Director, Quality Assurance Manager, and Operations Manager are responsible for ensuring the Project Manager has the appropriate resources available to respond to all client validation inquiries.

SCOPE

This policy is to be enforced and followed throughout the laboratory.

POLICY

1. The laboratory Project Manager receives data validation inquiries and will take one of the following courses of action:
 - 1.1 The Project Manager will forward the inquiry to the QA designee, who in turn will send the inquiry to the associated laboratory operation Group Leaders. If this option is chosen, the QA designee becomes responsible for tracking status and gathering all responses. Upon completion, the responses will be sent to the Project Manager. The Quality Assurance Manager and Operations Manager will be carbon copied on all email traffic.
 - 1.2. The Project Manager will forward the inquiry directly to the associated laboratory operation Group Leaders. The QA designee, Quality Assurance Manager, and Operations Manager will be copied on all email traffic. If this option is chosen, the Project Manager becomes responsible for tracking status and gathering all responses. Upon completion of the inquiry, the Project Manager must inform via email the QA designee, Quality Assurance Manager, and Operations Manager on the closure of the issue. If assistance is needed in assignment of duties, the Project Manager is to contact the Quality Assurance Manager for guidance.
2. The email to the Group Leaders should include, at a minimum, the following items:
 - a) Project Lot number and/or sample work order numbers, b) the specific question or original email from the client, and c) the “assignment of duties” based on the inquiry.
3. Upon receipt of a data validation inquiry, the Group Leaders must review the inquiry, and unless instructed otherwise by the Project Manager, either: a) email a detailed response within 24 hours that describes how the validation issue was resolved, or b) email a proposed turn-around time within 24 hours in which the issue will be addressed and completed. Emailed responses from the Group Leaders must be

presented in a manner that fully explains how the error occurred and the corrective action that was taken to resolve the issue. The Project Manager must be notified immediately of any circumstances or situations that may result in the delay of a response, and in turn, the Project Manager must update the client with the status of the inquiry.

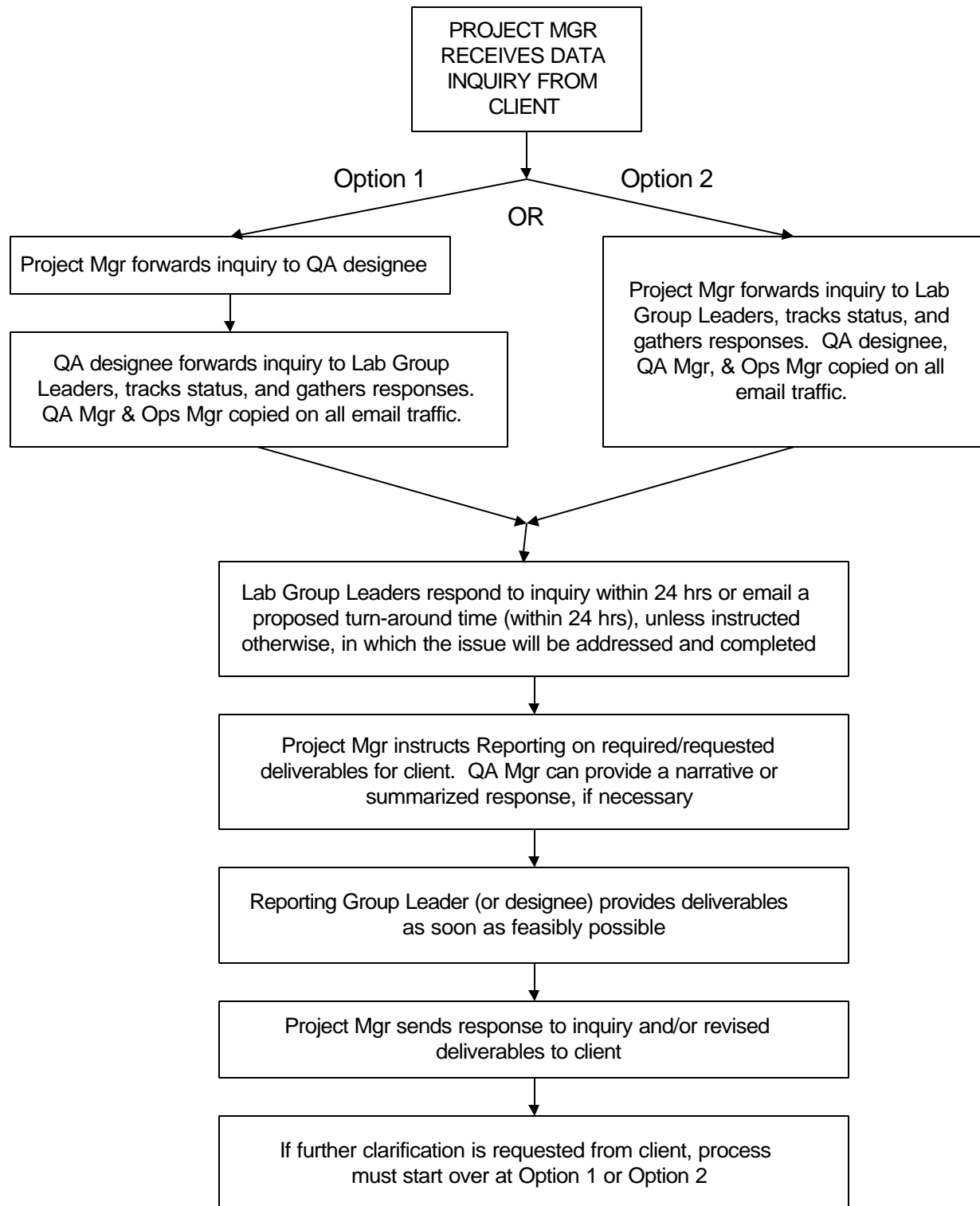
4. The Quality Assurance Manager will provide a recommended narrative change and/or a summarized response with corrective action noted if applicable. If the client requests further clarification or responses, the Project Manager must follow the aforementioned steps in order to resolve the issue. When all issues have been answered satisfactorily and the inquiry is closed, the Project Manager must provide the response to the client.
5. The Project Manager is responsible for instructing the Report Production Group Leader if reissued deliverables (reports, EDDs, CDs, etc.) are required. It is then the responsibility of the Report Production Group Leader to provide the requested deliverables and/or to post the electronic version of the deliverables in a public directory as soon as feasibly possible. Any inquiries about report production such as missing pages, extra copies, etc. are to be handled by the Report Production Group Leader.
6. The Quality Assurance Department is responsible for tracking all inquiries and reporting them on a monthly basis to management. Evaluation for any trends or system improvements must be performed on a monthly basis.
7. Refer to the Data Validation Flowchart for a visual description of the process.
8. Revision History

Historical File:

Revision 0: 01/23/02
Revision 1: 09/17/04
Revision 2: 10/29/04

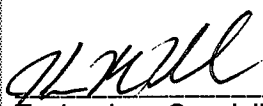
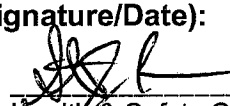


Revision 3: 08/03/07
Revision 4:

Data Validation Response Flowchart



Title: SAMPLE RECEIVING

[Method: None]

Approvals (Signature/Date):			
	<u>6/30/08</u>		<u>6/30/08</u>
Technology Specialist	Date	Health & Safety Coordinator	Date
	<u>6/30/08</u>		<u>6/30/08</u>
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-SC-0005, Rev 6.4, dated 10/02/06

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1. SCOPE AND APPLICATION

- 1.1. It is the responsibility of the Sample Receiving Department personnel to perform the procedures described herein in full compliance with this SOP.
- 1.2. It is the responsibility of the Laboratory Director, QA Manager, Operations Manager, and Departmental Supervisor of the facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable personnel to perform this SOP correctly.
- 1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

- 2.1. Not applicable

3. DEFINITIONS

- 3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

- 4.1. Not applicable.

5. SAFETY

- 5.1. Procedures shall be carried out in a manner that protects the health and safety of all TestAmerica associates.
- 5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves are worn for tasks that present a strong possibility of getting cut. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment (PPE) and precautions must be utilized. Disposable gloves that have been contaminated will be removed and discarded.
- 5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
Hydrochloric Acid	Corrosive Poison	5 ppm- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm- TWA 4 ppm- STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 mg/m ³ - Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m ³ - TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

- 5.4. Exposure to chemicals must be maintained as low as reasonably achievable. If samples are known to be hazardous, they are opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation, where possible. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

- 6.1. Thermometers
- 6.2. PPE such as gloves, lab coats, safety glasses, etc.
- 6.3. Utility knives
- 6.4. pH paper
- 6.5. Copier, printer, computer and label generator
- 6.6. Carts

7. REAGENTS AND STANDARDS

- 7.1. Not applicable

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Not applicable

9. QUALITY CONTROL

- 9.1. Nonconformance and Corrective Action
 - 9.1.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Not applicable

11. PROCEDURE

- 11.1. Receiving and Unpacking Samples
 - 11.1.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.
 - 11.1.2. The procedures listed in this document describe the responsibilities of Sample Receiving personnel in ensuring that data is transmitted correctly from the client to all personnel involved with sample analysis and review.

- 11.1.3. Samples typically arrive packed in coolers or foam boxes. Upon receipt, the coolers are triaged based on rush status (TAT: turn-around time) and expirable tests listed on the Chain-of-Custody (COC). Refer to Appendix I for a listing of expirable tests.
- 11.1.4. The following flagging system is used to show the priority level associated with triaged samples:
- 24 hour TAT – flag with red folder
 - 48 hour TAT – flag with blue folder
 - 72 hour TAT – flag with yellow folder
 - 1 week TAT – flag with green folder
 - Encore or low-level mercury samples – flag with orange paper with “Encore” or “LL Hg” written on it
- 11.1.5. If the COC lists expirable tests, the cooler is marked with a manila folder.
- 11.1.6. Rush and expirable coolers are to be unpacked first and logged as soon as possible. The lab groups are to be notified with any rush TAT requiring 72 hours or less. Samples requiring a standard TAT and/or no expirable tests are given lowest priority.
- 11.1.7. The following information is documented on the Cooler Receipt Form (CRF). Refer to Appendix II for a current version.
- 11.1.7.1. Client name
- 11.1.7.2. Project name or number (if known)
- 11.1.7.3. Signature of Sample Receiving Technician
- 11.1.7.4. Date cooler received and opened
- 11.1.7.5. Method samples were received (overnight courier, client drop-off, or other means)
- 11.1.7.6. Cooler ID number (if known)
- 11.1.7.7. Type of container received (TestAmerica cooler, client cooler, foam box)
- 11.1.7.8. Presence of the custody seals on the outside of the cooler
- 11.1.7.9. Presence of custody seals on bottles
- 11.1.7.10. Presence of the custody papers (i.e., COC)

- 11.1.7.11. Verification that custody papers were properly filled out (ink, signed, match labels)
- 11.1.7.12. Verification that custody papers were relinquished by the client
- 11.1.7.13. Presence of a packing slip
- 11.1.7.14. Presence and type of packing material information
- 11.1.7.15. Cooler temperature upon receipt. The temperature of the cooler is taken by an IR gun. The IR gun is directed at the label (minimizes laser reflection) of a sample bottle or temperature vial, whichever best reflects the cooler temperature. According to federal regulations, the temperature upon receipt should be $\leq 6^{\circ}\text{C}$. The Project Manager (PM) is contacted (and the anomaly is narrated) when the temperature is $> 6^{\circ}\text{C}$ or if samples are frozen.
- 11.1.7.16. Condition of bottles upon receipt (good condition, broken, etc.)
- 11.1.7.17. Complete bottle labels (date, time, client ID)
- 11.1.7.18. Information on bottle labels and tags agree with custody papers
- 11.1.7.19. Verification that the correct bottles were received for the tests indicated (refer to Appendix III)
- 11.1.7.20. Volatile (VOA) vials were checked for the presence of air bubbles. If vials have bubbles exceeding 6 mm in diameter, it is narrated and the Project Manager (PM) is contacted.
- 11.1.7.21. Verification that sufficient amount of sample was received in order to perform the tests listed on the COC.
- 11.1.7.22. Verification of pH for preserved samples (except Volatiles and TOC) upon receipt. This is done by removing sample lids and using a droplet of sample from in the lid to test the pH. The pH's are then recorded on the CRF. The pH paper strips are then discarded.
 - 11.1.7.22.1. Purchased prepared vials of preservatives (refer to Appendix IV) are used if samples are not at the correct pH. The pH is adjusted by adding the appropriate preservative in 5 mL increments up to a maximum of 20 mL per liter of sample or unless there is a reaction. The pH adjustment and final pH are noted on the CRF. The Lot Number of the pre-made preservative is noted on the CRF. It is the responsibility of the Sample Receiving group to change the lot number when a new shipment arrives and to document when a new box is opened.

11.1.7.22.2. The concentrations of the preservatives used are as follows:

4N Sodium Hydroxide

1N Zn Acetate

1:1 HCL (18%)

1:4 HNO₃ (18%)

1:2 H₂SO₄ (33%)

11.1.7.23. The presence or absence of a Trip Blank and if VOA samples were received.

11.1.7.24. Documentation (name of PM, date, name of Sample Receiving technician, method used to contact PM) that the PM was notified of any anomalies associated with receipt of a project.

11.1.8. The Sample Receiving technician removes all sample containers. Any broken, leaking, or dirty sample containers are to be placed inside the fume hood. Dirty sample containers are to be cleaned appropriately, so as not to contaminate the sample storage area.

11.1.9. Coolers emitting strong vapors/fumes when opened will be taken to the High Hazard Room and unpacked in either of its hoods.

11.1.9.1. Any problems concerning exposure while unpacking samples must be immediately reported to the Group Leader and/or the EH&S Coordinator.

11.1.10. Volatile sample(s) suspected (e.g., odor) or known (client information or site history) to be high in volatile concentration is stored in a separate designated volatile area.

11.1.11. The Sample Receiving technician examines all documents and compares information on the sample container labels to the COC. All discrepancies are documented on the CRF.

11.1.12. Problems or discrepancies that compromise sample integrity (such as limited sample volume, sample identification which cannot be determined from the COC, incorrect pH levels, or preservatives, if known, or broken leaking samples) are reported to the PM. The PM will advise Sample Receiving on how to proceed.

11.1.13. Expirable tests (hold time 48 hours or less) must be written on the top of the bottles from which they are to be analyzed. If more than one method exists for the analysis, the method must also be written.

11.1.13.1. Sample containers are placed in the red bin designated for expirables in the Sample Receiving room. The work order or sample ID must be recorded on the expirable logsheet along with the record of the test to be run, special method, if necessary, and the initials of the person relinquishing the sample. The Wet Chem Lab group checks this bin throughout the day, and is responsible for signing out the sample container when they take it.

11.1.14. Samples requiring a Total Solid (TS) result are split off for analysis. Splitting these samples is the responsibility of the Sample Receiving group. A small representative portion (approx.

5-10 grams) from each sample is placed into a small plastic snap-top container designated for the TS analysis. If the TS container is not labeled with a LIMS label when the aliquot is removed, the TS container must be labeled with handwritten client ID. Containers designated for volatiles analyses (VOC) must not be opened in Sample Receiving. If only one container is received and volatiles are requested, the Receiving group must **not** split off because of possible contamination or possible lost volatiles. An empty TS container with a LIMS label is given to the VOC Analysis group for each solid. The VOC group will aliquot for the TS when they open the container for analysis. The TS plastic containers are placed in baggies according to lot number, and are stored in a box inside the walk-in cooler door.

11.1.15. When samples need to be composited, the following procedure is followed, unless there are specific instructions from the client.

11.1.15.1.1. Equal aliquots are weighed from each container and mixed thoroughly and transferred to a new container.

11.1.15.1.2. The amount aliquoted (in grams) is recorded on the Cooler Receipt Form.

11.1.16. The Sample Receiving technician who unpacked the cooler must sign the COC in the appropriate place.

11.1.17. Samples received after hours are signed for by a TestAmerica North Canton employee and placed in the walk-in cooler to be processed the following business morning.

11.2. Sample Log-In

11.2.1. Samples are logged in LIMS after unpacking has been completed, and all anomalies have been documented.

11.2.2. All COCs are photocopied, and given to the associated PM, who in turn, provides the appropriate quote to be used for log-in.

11.2.3. A quote must reflect what is on the Chain-of-Custody. Any discrepancies must be resolved by the PM. Projects are generally not logged until the PM provides a quote. However, if it is a routine project and the quote is known, log-in can proceed.

11.2.4. In the event a project cannot be logged due to discrepancies, all associated paperwork is placed into a black folder. All samples and the black folder are put into cold storage. These projects are recorded on the dry-erase board posted on the walk-in cooler door.

11.2.5. Log-in is initiated by retrieving the paperwork (including a photocopy of the COC or an email from the PM, which lists the TAT and quote number) from the unpacked projects on the counters. The following information is included at log-in: analytical due date, report due date, type of package (expanded deliverable or standard), client sample ID, sample collection date and time, sample matrix (example: WW = wastewater, WG = groundwater, SO = solid), sample type (example: Normal = N1, Trip Blank = TB, Rinse Blank = RB1), and the analyses requested on the COC.

11.2.5.1. A unique lot number is generated for each project that is logged. The lot number is nine characters in length, and is based on the date of receipt. For example, Lot No. A8C010121 is described as follows:

A - TestAmerica location where the samples were received
(A = North Canton, C = Pittsburgh, etc.)

8 - Last digit of the year (i.e., 2008)

C - Month (i.e., A = January, B = February, C = March)

01 - These two numeric characters identify the day of the month--in this case, the first day of the month.

0121 - These four numeric characters are the sequential assignment of numbers specific to each lot received. Each day, the first lot logged receives the number "0101"; the second lot receives the number "0102", etc.

As each sample is logged, individual *sample numbers* are created in sequential order, noted as the three-digit suffix following the lot number.

For example, four samples were received on May 10, 2008 for PCB analysis with the following client IDs: AB100, AB101, AB102, AB103.

<u>Client ID</u>	<u>Lot No. – Sample No.</u>	<u>Analysis</u>
AB100	A8E100101-001	PCB
AB101	A8E100101-002	PCB
AB102	A8E100101-003	PCB
AB103	A8E100101-004	PCB

11.2.6. If a client requests an MS/MSD for a sample(s), it is denoted in LIMS by an "S" for the MS and a "D" for the MSD.

11.2.7. Work Order Numbers: As each parameter is logged, a random unique 8-digit Work Order number is generated by LIMS. For example, Work Order No. A5WE1-1-1C is described as follows:

A5WE1 - The first five characters of the Work Order number identifies each unique sample.

1 - The "modifier" indicates the run number. This number will always be a "1" at log-in. However, additional modifier numbers may be generated by the analytical lab groups when necessary (example: for a re-extract).

1C - The "suffix" identifies a specific test for each sample, but is unique to the test being performed on each sample.

11.2.8. Each sample container representing the same sample (as indicated by the client ID) will have the same 5-digit Work Order number, along with a 3-digit suffix that corresponds to the bottle

count (example: -001, -002, -003, etc.). For example, if five containers are submitted for the same sample and the LIMS-generated Work Order number is CREE4, LIMS will generate five labels--CREE4-001, CREE4-002, CREE4-003, CREE4-004, CREE4-005. These numbers are used to track sample containers through the analytical process.

11.3. Labeling

11.3.1. After log-in is completed, the sample containers are labeled. All labels contain the following information: lot number, sample number, Work Order number, bottle count number, number and type of bottles, client sample ID, client code, date/time of sample collection, and the type of project (example: RCRA) (refer to Appendix V). Labeling samples is a critical step in the log-in process. Care must be taken to ensure client sample ID's and lab ID's match, sample storage locations are correct, the container count and type is correct. Mislabeling can result in incorrect text being run on inappropriate samples.

11.3.1.1. Labels that read "Caution-Use Hood!" shall be affixed to all containers for a given sample that are thought to be a safety hazard (for example, high in contaminants, flammable, etc.), or known to emit noxious fumes or odors. The Sample Receiving group is notified of potential hazards by the PM, COC, quote, or client.

11.3.1.1.1. Samples that are known or expected to contain high concentration of Cyanide (250 ppm or more) or Sulfide (500 ppm or more) need to be unpacked in a fume hood. The Sample Receiving group must put a special sticker on these sample bottles indicating to the Lab Groups that the samples are high in either Cyanide or Sulfide so the Lab Groups can take the necessary safety precautions.

11.4. Storage

11.4.1. Once all sample containers have been properly labeled, the Sample Receiving technician will place the samples into the proper storage locations. These locations are as follows:

- Organic extractable samples are placed in the walk-in refrigerators located in Sample Receiving.
- Volatile samples are stored in the double-door refrigerator located in the Sample Return area.
- Samples known or suspected to be hazardous are stored in a cage in the walk-in cooler, in a fume hood in the High-Hazard Room, and/or in a fume hood in the Volatile rooms.
- Inorganic samples are stored in the walk-in refrigerators located in Sample Receiving.
- Preserved metal samples are placed in a non-refrigerated room. Metals samples that need to be lab filtered and/or preserved are stored in the walk-in cooler. When advised of a Navy project, metal samples are stored in the walk-in cooler.

11.5. Paperwork

- 11.5.1. All paperwork (COC, CRF, LIMS log-in sheets) associated with a project is clipped together and referred to as a Summary.
- 11.5.2. For clients who request a show of sample transfer from sample receipt to storage, a sample control record is printed (see Appendix VI). TestAmerica LIMS will generate this record for Expanded Deliverable designated samples. This record is referred to as an Internal Chain-of-Custody (COC).
- 11.5.3. The Internal COC is attached to the Summary.

11.6. Bottle Returns

- 11.6.1. After analysts have retrieved and analyzed samples, the containers are returned to Sample Receiving for storage and/or disposal documentation.
- 11.6.2. The Sample Return area is located in a hallway behind the walk-in-cooler.
- 11.6.3. A "Sample Custodian Removal Request Form" accompanies the samples (see Appendix VII).
- 11.6.4. The Sample Receiving technician records the return on the Removal Request Form and re-enters custody of the bottles to the Sample Receiving area. The Sample Receiving technician initials and dates the Return form.
- 11.6.5. When sample bottles are consumed in the analysis process, the empty container is returned. The letter "C" is marked on the Removal Request Form beside the appropriate sample to indicate a consumed sample.
- 11.6.6. Samples returned after Sample Receiving Dept. has left for the day are placed in the walk-in cooler by the analyst. Sample Receiving records as received on the next working day.
- 11.6.7. All Removal Request Forms remain in the Sample Return area for approximately one month. After this time, the forms are boxed and stored for seven years.

11.7. Subcontracting of Samples

- 11.7.1. Samples that are logged for parameters performed at other laboratories are subcontracted to these facilities (including other TestAmerica labs).
- 11.7.2. A Sample Analysis Requisition (SAR) is printed for these samples upon completion of the log-in process (see Appendix VIII).
- 11.7.3. The SAR contains information necessary for sample analysis. The original form accompanies the samples to the subcontracted laboratory, and a copy is attached to the Summary. The SAR form must have a relinquished signature with a date and time. Any additional information necessary for sample analysis must be handwritten on the form (e.g. list of

compounds, homogenizing of samples, limited quantity, etc.). In order to track subcontracted samples, the lab Purchase Order number on the SAR form must be recorded in the subcontracted sample PO book located in the Sample Receiving log-in area. (Appendix IX).

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Training Qualifications

13.1.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

13.1.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

13.1.3 The Group/Team Leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.1.4 The only personnel authorized to execute this SOP are those in the Sample Receiving Dept.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention".

15.2. PPE required for purging sample wastes include a lab coat, face shield, and gloves.

15.3. All samples may be disposed of 30 days after the report date except for those samples associated with special client retention. Shelves are purged in chronological order. All lots on a specific shelf must be noted. If a lot can be disposed, a disposal date must be recorded.

15.4. Disposal dates are recorded on this print-out. When clearing shelves, samples that cannot be disposed of because of special client retention are boxed or stored on carts. Stored samples must have a specific

date listed in which samples can be disposed of, or a note that indicates “SAVE” and client name or reason.

- 15.5. A print-out of all lots assigned to certain shelves in each storage location can be obtained from LIMS.
- 15.6. Solid samples that are non-regulated waste are placed in a cubic yard container for disposal.
- 15.7. Regulated solid waste is placed in the “MIXED WASTE” container.
- 15.8. Water samples designated for disposal are placed on carts and disposed of in Sample Receiving. Acidified samples are poured into a drum and neutralized under a fume hood in Sample Receiving.
- 15.9. Solvent waste must be disposed of in clearly labeled waste cans.

16. REFERENCES

16.1 References

16.1.1 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.2 TestAmerica Corporate Safety Manual, M-E-0001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.2 Associated SOPs and Policies, current version

16.2.1 QA Policy, QA-003

16.2.2 Supplemental Practices for DoD Project Work, NC-QA-0016

16.3 Revision History

<u>Historical File:</u>	Revision 6: 09/02/99	Revision 6.3: 04/20/04
	Revision 6.1: 06/14/01	Revision 6.4: 10/02/06
	Revision 6.2: 10/06/03	

17. MISCELLANEOUS

17.1 Appendices

17.1.1 Appendix I – Example of Expirables

17.1.2 Appendix II – Example of Cooler Receipt Form

17.1.3 Appendix III – Example of Sample Containers, Preservatives, Holding Times

17.1.4 Appendix IV – Example of Preservatives

17.1.5 Appendix V – Example of Label

17.1.6 Appendix VI – Example of Internal Chain-of-Custody

17.1.7 Appendix VII – Example of Sample Custodian Removal Request

17.1.8 Appendix VIII – Example of Sample Analysis Requisition

17.1.9 Appendix IX – Example of Sub-Work Tracking

Appendix I – Example of Expirables

TESTAMERICA NORTH CANTON EXPIRABLES

24-HOUR HOLD TIME:

pH – WATER (9040/4500/150.1—must be on bottle)

*Cr⁺⁶ – WATER (7196/3500—must be on bottle)

FERROUS IRON

RESIDUAL CHLORINE

DISSOLVED OXYGEN (DO)

CORROSIVITY – WATER

48-HOUR HOLD TIME:

BOD/CBOD

*NO₂ – IC (300/9056) or TRACCS (353)

*NO₃ – IC (300/9056) or TRACCS (353)

*OPO₄ – IC (300/9056) or MANUAL (365/4500)

ALKALINITY – TOTAL, CARB, BI-CARB, HYDROXIDE

TURBIDITY

CORROSIVITY – SOLID

SETTLEABLE SOLID

*CR⁺⁶, NO₂, NO₃, AND OPO₄ ARE VERY IMPORTANT.
THESE SHOULD BE DONE FIRST!!!

Appendix III – Example of Sample Containers, Preservatives, Holding Times

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative _{1,2}	Volume	Parameter	Container	Preservative _{1,2}	Volume
601/602	G	HCl	3x40 mL	PCB	G	None	2 L
Acidity	P	None	250 mL	Pesticides	G	None	2 L
Alkalinity (Sep)	P	None	250 mL	Pesticides + PCBs	G	None	2 L
Amenable Cyanide	P	NaOH	250 mL	pH	P	None	50 mL
Ammonia Nitrogen (NH ₃)	P	H ₂ SO ₄	500 mL	Phenols	G	H ₂ SO ₄	1 L
Asbestos	P	None	250 mL	PNA/PAH	G	None	2 L
Bicarbonate	P	None	250 mL	R. Chlorine	P	None	100 mL
BNA + Dioxin	G	None	2 L	Radiological Alpha, Beta, Radium	P	HNO ₃	4 L
BNAs	G	None	2 L	Reactive Cyanide	P	None	1 L
BOD	P	None	250 mL	Reactive Sulfide	P	None	1 L
Bromide (Br)	P	None	250 mL	Settleable Solids	P	None	1L
BTEX & MTBE	G	HCl	3x40 mL	Silica	P	None	250 mL
BTEX 8021	G	HCl	3x40 mL	Sulfate	P	None	250 mL
Carbonaceous BOD	P	None	250 mL	Sulfide	P	Zn Acetate & NaOH	1 L
Carbonate	P	None	250 mL	Sulfite	P	None	250 mL
Chemical Oxygen Demand	P	H ₂ SO ₄	250 mL	Surfactants (MBAS)	P	None	250 mL
Chloride (Cl)	P	None	250 mL	T. Coliform	P	None	125 mL
Chromium, ⁶⁺	P	None	250 mL	TDS	P	None	250 mL
COD	P	H ₂ SO ₄	250 mL	TKN	P	H ₂ SO ₄	1L
Color	P	None	50 mL	TON	P	H ₂ SO ₄	1 L
Conductivity	P	None	250 mL	Total Cyanide	P	NaOH ³	250 mL
Corrosivity	P	None	250 mL	Total Organic Carbon (TOC)	G	HCl	2 x40 mL
Dissolved Metals*	P	HNO ₃	1 L	Total Organic Halogens	G	H ₂ SO ₄	250 mL
Dissolved Oxygen	G	None	300 mL	Total Phosphorus	P	H ₂ SO ₄	250 mL
Elemental PO ₄	G	None	250 mL	Total Solids	P	None	250 mL
Fecal Coliform	P	None	125 mL	TPH - Diesel (Ext.)	G	None	2 L
Flashpoint	G	None	100 mL	TPH - Gasoline (P&T)	G	HCl	2x40 mL
Fluoride	P	None	250 mL	TPH-GC	G	None	2 L
Formaldehyde	G	None	500 mL	TRPH - IR 418.1	G	HCl	2 L
Free Cyanide	P	NaOH	250 mL	TSS	P	None	250 mL
Glycols 8015	G	None	2x40 mL	Turbidity	P	None	250 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative _{1,2}	Volume	Parameter	Container	Preservative _{1,2}	Volume
Hardness	P	HNO ₃	250 mL	TVS	P	None	250 mL
Herbicides	G	None	2 L	VOC	G	HCl	3x40 mL
Metals	P	HNO ₃	1 L	VOC 601	G	HCl	3x40 mL
Nitrate	P	None	250 mL	VOC 602	G	HCl	3x40 mL
Nitrate/Nitrite	P	H ₂ SO ₄	250 mL	VOC 624	G	HCl	8x40 mL
Nitrite	P	None	250 mL	VOC 624	G	HCl	3x40 mL
Oil & Grease	G	H ₂ SO ₄	1 L	VOC 8260	G	HCl	3x40 mL
OPPs	G	None	2 L	VOC and VOA	G	HCl	3x40 mL
Orthophosphate	P	None	250 mL				

Notes:

* Filtered in field

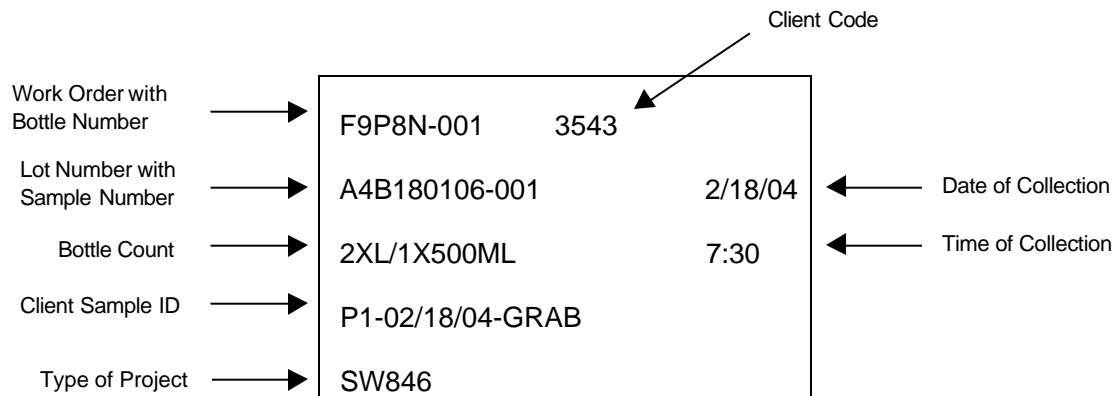
¹ HCl, HNO₃, and H₂SO₄ to pH < 2. NaOH to pH > 12² Temperature = ≤ 6°C except for aqueous metals

Appendix IV – Example of Preservatives

PRESERVATIVES

NITRIC ACID	< 2	HNO ₃	Add Nitric Acid to Contents After Filling
SULFURIC	< 2	H ₂ SO ₄	Add Sulfuric Acid to Contents After Filling
SODIUM HYDROXIDE	> 12	NAOH	Add Sodium Hydroxide to Contents After Filling
HYDROCHLORIC ACID	< 2	HCl	Add Hydrochloric Acid to Bottle Prior to Filling
			Add Zinc Acetate to Contents After Filling
ZINC ACETATE/SODIUM	> 9	ZNACE/NAOH	Add Sodium Hydroxide to Contents After Filling

WI-NC-004A-022007

APPENDIX V – Example of Laboratory-Generated Bottle Label

Appendix VI – Example of Internal Chain-of-Custody

TestAmerica Laboratories, Inc.

Sample Control Record

Client:

Lot #:

Case Number/SDG:

Storage Location:

Laboratory Sample ID	Transferred By	Date	Entered	Removed	Reason	Date Returned

Appendix VII – Example of Sample Custodian Removal Request

PSR024 5/23/08 6:47:31 MT SAMPLE CUSTODIAN REMOVAL REQUEST PAGE 001

REQUESTED BY:

METHOD: B2 Fluoride (4500F-C, ISE, SM18)

STORAGE LOCATION	WORK ORDER #	PICKED CNTR#	CONTROL #	CLIENT #	ANALYSIS	LOTID	SMP#	SPX	MATRIX DESCRIPTION	QTY RCVD	QTY REQD
C36 S31 MS	KM6HD-1-AQ	007	348422	001610	I-88-B2 ASE140184	001			WATER	8	1
C36 S31 MS	KM6HX-1-AQ		348423	001610	I-88-B2 ASE140184	002			WATER	8	1
C36 S31 MS	KM6HL-1-CL		348424	001610	I-88-B2 ASE140184	003			WATER	24	1
C36 S31 MS	KM6HN-1-AQ		348425	001610	I-88-B2 ASE140184	004			WATER	8	1
C36 S31 MS	KM6HP-1-AQ		348426	001610	I-88-B2 ASE140184	005			WATER	8	1
C36 S31 MS	KM6HR-1-AQ		348427	001610	I-88-B2 ASE140184	006			WATER	8	1
C36 S31 MS	KM6HV-1-AQ		348428	001610	I-88-B2 ASE140184	008			WATER	8	1
C36 S31 MS	KM6HX-1-AQ		348429	001610	I-88-B2 ASE140184	009			WATER	8	1
C54 S35 MS	KM6DL-1-AQ		348430	001610	I-88-B2 ASE150313	002			WATER	8	1
C54 S35 MS	KM6DP-1-CL		348431	001610	I-88-B2 ASE150313	003			WATER	24	1
C54 S35 MS	KM6DW-1-AQ		348432	001610	I-88-B2 ASE150313	004			WATER	8	1
C54 S35 MS	KM6DX-1-AQ		348433	001610	I-88-B2 ASE150313	005			WATER	8	1
C54 S35 MS	KM6D2-1-AQ		348434	001610	I-88-B2 ASE150313	006			WATER	8	1
C54 S35 MS	KM6D3-1-AQ		348435	001610	I-88-B2 ASE150313	007			WATER	8	1
C54 S35 MS	KM6DC-1-AQ		348436	001610	I-88-B2 ASE150313	009			WATER	8	1
C54 S35 MS	KM6DF-1-AQ		348437	001610	I-88-B2 ASE150313	010			WATER	8	1

RELINQUISHED BY

RECEIVED BY

DATE/TIME

***** END OF REPORT *****

Appendix VIII – Example of Sample Analysis Requisition

TestAmerica Laboratories, Inc.
SAMPLE ANALYSIS REQUISITION

Laboratory: (Subcontracting Lab)

Report Package: (Type of Report)

Lab Request: (Sample Rcvg. P.O.)

Need Analytical Report: (Date)

Client Code:

Project Manager:

<u>Sample I.D.</u>	<u>W.O. No.</u>	<u>Client Sample ID</u>	<u>Sampling Date</u>	<u>Analysis Required</u>
Example : A8B010350	F9DED	MW-6	2008-01-31 11:12	WATER, 6010B, Total Lithium - (Subcontr. Lab)

Please use Client Sample ID for report
Call (Project Manager) with questions at 330-497-9396
At TestAmerica North Canton Laboratory

Need detection limit and analysis date included in report.

Shipping Method: **FEDEX**

Please send a signed copy of this form with the report at completion of analysis.

Relinquished by: _____ Date/Time: _____

Relinquished by: _____ Date/Time: _____

Received for lab by: _____ Date/Time: _____

Appendix IX – Sub-Work Tracking

TestAmerica North Canton

Sub-Work Tracking

Tracking No.	Client Code	Lot Number	No. of Samples	Analysis	Lab Sent To	Date Out	Shipment Method	Initials

Cover Page:

Quality Assurance Manual

TestAmerica North Canton
4101 Shuffle Street NW
North Canton, OH 44720
Phone: 330-497-9396
Fax: 330-497-0772
www.testamericainc.com

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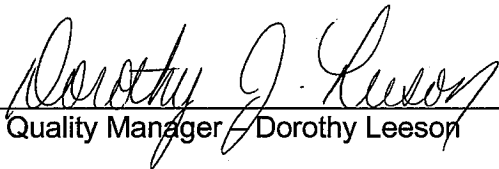
**Quality Assurance Manual
Approval Signatures**



Laboratory Director – Opal Davis-Johnson

12/10/07


Date



Quality Manager – Dorothy Leeson

12/10/07

Date



Technical Director – Dr. Mark Bruce

12/10/07

Date

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SOPs AND POLICIES REFERRED TO IN THE QA MANUAL

SOP/Policy Reference	Title
CA-C-S-001	Work Sharing Process
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CA-L-S-002	Subcontracting Procedures
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-003	Management of Change Procedure
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-005	Calibration Curves (General)
CA-T-P-001	Qualified Products List
CORP-MS-0001NC	GC/MS Analysis Based on Method 8270C,
CORP-MS-0002NC	Determination of Volatile Organics by GC/MS based on Method 8260B & 8260A).
CORP-QA-0010	Nonconformance and Corrective Action System
CORP-QA-0013	Employee Orientation and Training
CW-F-P-002	Authorization Matrix
CW-F-S-004	Controlled Purchases Policy
CW-L-P-001	Record Retention
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
NC-QA-0012	Shipping Department
NC-QA-015	Equipment Monitoring and Thermometer Calibration
NC-QA-0017	Standards and Reagents
NC-QA-0018	Statistical Evaluation of Data and Development of Control Charts
NC-QA-0019	Records Information Management
NC-QA-0020	Laboratory Holding Blanks
NC-QA-0021	Evaluation of Method Detection Limits for Chemical Tests

NC-QA-0027	Preparation and Management of Standard Operating Procedures
NC-SC-0005	Sample Receiving and Sample Control
NC-SC-0006	Sample Procurement Protocol
QA-003	TestAmerica North Canton Quality Control Program
S-C-002	Complaint Handling and Service Recovery
S-Q-001	Document Control
S-Q-004	Acceptable Manual Integration Practices

SECTION 3

INTRODUCTION (NELAC 5.1 - 5.3)

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

The TestAmerica North Canton Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica data quality goals. Each TestAmerica laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (1999). In addition, the policies and procedures outlined in this manual are compliant with the various accreditation and certification programs listed in Appendix 7. The relevant NELAC section is included in the heading of each QAM section.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA, March 1979.
- EPA SW-846, *Test Methods for the Evaluation of Solid Waste*, 3rd Edition, September 1986; Update I, July 1992; Update II, September 1994; and Update III, December 1996.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by TestAmerica North Canton conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 6 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

TestAmerica analyzes thousands of environmental and industrial samples every month. Sample matrices vary among effluent water, groundwater, hazardous waste, sludge, wipes, and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and

tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested water, industrial waste, and soil methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by TestAmerica North Canton shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, the Quality Assurance (QA) Manager, and the Technical Director. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

- Specific requirements delineated in project plans may supersede general quality requirements described in this manual. Ohio VAP requirements are listed throughout the document.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

The manual is reviewed annually by the QA Manager and laboratory personnel to assure that it reflects current practices and meets the requirements of TestAmerica North Canton clients and regulators. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. The updates will be reviewed by the QA Manager, Laboratory Director/Manager, Technical Director(s), relevant operational staff and Corporate Quality Assurance (if a change is made to the Corporate template) and then formally incorporated into the document in periodic updates. The QAM is based on a Corporate QAM Template that is prepared and approved by the Chief Operating Officers (COOs) and Corporate Quality Assurance. This template is reviewed annually by the COOs, Corporate Quality, and each laboratory. Necessary changes are coordinated by the Vice President of Quality and Environmental Health & Safety (EHS) and distributed to each laboratory for inclusion in the laboratory specific QA Manuals.

Policies in the QAM that require immediate attention may be addressed through the use of Corporate QA/QC Policy Memoranda. QA/QC Policy Memoranda are published from time to time to facilitate immediate changes to QA/QC Policy. QA/QC Policy Memoranda supersede the QAM and all other SOPs (refer to Section 5.3). All policy memoranda are dated, archived and distributed by their placement into the front of the QAM between the signature page and Section 2. At a minimum, each policy memorandum is approved by the same authorized signatories as shown on the cover page of the QA Manual. In addition, Corporate QA/QC Policy

Memoranda are signed by the COOs and VP of Quality and EHS. The QA/QC Policy Memoranda are incorporated into the QAM during the periodic updates. Policy memorandum may also include an expiration date if appropriate. An example format can be found in Figure 3-1. A similar procedure is followed for local laboratory changes.

3.4.2 Control

This manual is considered confidential within TestAmerica and may not be altered in any manner by other than a duly appointed representative from TestAmerica. If the document has been provided to external users or regulators, it is for the exclusive purpose of reviewing TestAmerica North Canton quality systems and shall not be used in any other way without the written permission of an appointed representative of TestAmerica. The procedure for control of distribution is incorporated by reference to SOP S-Q-001, Document Control.

The order of precedence in the event of a conflict between policies is outlined in Section 5.3 of this Quality Assurance Manual.

Figure 3-1.

Example - Format for a QA/QC Policy Memorandum

TestAmerica North Canton	
SOP/LQM CHANGE FORM	
SOP/LQM NUMBER:	
TITLE:	
SECTION(S) AFFECTED BY CHANGE:	
REASON FOR ADDITION OR CHANGE:	
CHANGE EFFECTIVE FROM: (DATE):	
CHANGE OR ADDITION:	
SUBMITTED BY/DATE:	
*APPROVED BY:	
Technical Reviewer Signature	Date
Environmental Health & Safety Signature	Date
QA Signature	Date
Technical Director (if applicable)	Date
Laboratory Director Signature	Date

SECTION 4

ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

4.1 OVERVIEW

TestAmerica North Canton is part of a national network of laboratories known as TestAmerica. This Quality Assurance Manual (QAM) is applicable to the TestAmerica North Canton laboratory only.

**TestAmerica North Canton Laboratory
4101 Shuffle Drive NW
North Canton, OH 44720
EPA I.D. Code No. 9503**

The Corporate organization chart can be found in Figure 4-1 and the laboratory organization chart can be found in Appendix 2. The locations of other TestAmerica labs are as follows:

Aerotech Environmental Laboratories (AEL)
TestAmerica Anchorage
TestAmerica Austin
TestAmerica Buffalo
TestAmerica Buffalo Grove
TestAmerica Burlington
TestAmerica Cedar Falls
TestAmerica Chicago
TestAmerica Colorado Springs
TestAmerica Connecticut
TestAmerica Corpus Christi
TestAmerica Dayton
TestAmerica Denver
TestAmerica Edison
TestAmerica Honolulu
TestAmerica Houston
TestAmerica Irvine
TestAmerica King of Prussia
TestAmerica Knoxville
TestAmerica Los Angeles
TestAmerica Mobile
TestAmerica Morgan Hill
TestAmerica Nashville
TestAmerica New Orleans
TestAmerica Ontario
TestAmerica Orlando
TestAmerica Pensacola
TestAmerica Phoenix
TestAmerica Pittsburgh

TestAmerica Portland
TestAmerica Richland
TestAmerica San Francisco
TestAmerica Savannah
TestAmerica Seattle
TestAmerica Spokane
TestAmerica St. Louis
TestAmerica Tacoma
TestAmerica Tallahassee
TestAmerica Tampa
TestAmerica Valparaiso
TestAmerica Watertown
TestAmerica West Sacramento
TestAmerica Westfield

4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of TestAmerica North Canton. All employees have access to the QAM and are responsible for knowing the content of this manual and upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs.

4.2.2 Chairman/Chief Executive Officer (CEO)

The Chairman/CEO is the Chairman of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the President/CEO of the Analytical Division, the Chairman/CEO establishes the overall quality standard and data integrity program for the company, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.3 President/Chief Executive Officer (CEO)

The President/CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the Chairman/CEO, the President/CEO establishes the overall quality standard and data integrity program for the Analytical Division, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.4 Chief Operating Officer (COO) – East and West

The COOs serve as the ranking executives for all respective analytical laboratory operational functions and report to the President/CEO of the Analytical Division. They are responsible for the daily management of all analytical laboratories, long-term planning and development of technical policies and management plans. They ensure the attainment of corporate objectives through the selection, development, motivation, and evaluation of top management personnel. The COOs approve all operating budgets and capital expenditures. The COOs sign-off on the final QAM template that contains company policies for implementing the Quality Program.

4.2.5 General Manager (GM)

Each GM reports directly to a COO. Each GM has full responsibility for the overall administrative and operational management of their respective laboratories. The GM's responsibilities include allocation of personnel and resources, long-term planning, setting goals, and achieving the financial, business, and quality objectives of TestAmerica. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews. The GM is also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.6 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President of QA/EHS reports directly to the Chairman/CEO. With the aid of the Analytical Division and Non-Analytical Division Senior Management Teams, Laboratory Director/Managers, Quality Directors, EHS Directors, QA Managers and EHS Coordinators, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and Environmental, Health and Safety Program within TestAmerica. Additional responsibilities include:

- Review of QA/QC aspects of Corporate SOPs, national projects and expansions or changes in services.
- Coordination/preparation of the Corporate QAM Template that is used by each laboratory to prepare its own laboratory-specific QAM.
- Maintenance of Corporate Policies, Quality Memorandums and SOPs. Maintenance of data investigation records that are reported to Corporate Management.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the Analytical Division and a summary of any quality related initiatives and issues.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

4.2.7 Quality Directors (Corporate)

The Quality Directors report to the VP-QA/EHS. Together with the VP-QA/EHS, the Quality Directors have the responsibility for the establishment, general overview and maintenance of

the Analytical Division's Quality Assurance Program within TestAmerica. The Quality Directors are responsible for:

- Oversight of the QA/QC programs within each laboratory. This includes a final review of each laboratory-specific QAM and receipt of each laboratory's QA monthly report.
- Review of QA/QC aspects of national projects.
- Assistance with certification activities.

4.2.8 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – VP-QA/EHS and VP-Client and Technical Services. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the CEOs, COOs, Laboratory Director/Manager or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.9 Vice President of Client and Technical Services

The Vice President (VP) of Client and Technical Services is responsible for offerings to clients including risk management, technical assistance, legal compliance and contract administration. The VP of Client and Technical Services provides support and direction to the Managers of these areas, and supports the COOs in decisions regarding long term planning, resource allocation and capital expenditures.

4.2.10 Director of Technical Services

The Director of Technical Services is responsible for establishing, implementing and communicating TestAmerica's Analytical Division's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

4.2.11 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica Information Technology (IT) Policies, SOPs, and Manuals. Other responsibilities include coordinating new technologies; development of electronic communication tools such as TestAmerica intranet and internet sites; ensuring data security and documentation of software; ensuring compliance with the NELAC standard; and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

4.2.12 Environmental Health and Safety Directors (EHSDs) (Corporate)

The EHSDs report directly to the VP-QA/EHS. The EHSDs are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.13 Laboratory Director

- Reports directly to the Regional General Manager
- Responsible for implementation and adherence by lab staff to the TestAmerica North Canton QAM and all policies and procedures within the laboratory
- Has signature authority for QAM, policies, SOPs, and contracts (as detailed in TestAmerica policy)
- Assesses the effectiveness of the QAM within the operation
- Maintains adequate trained staff documented on organization charts
- Responsible for implementing internal/external audit findings corrective actions

4.2.14 Quality Assurance Manager

- Reports directly to the Laboratory Director and, for all QA matters, to the Corporate QA Director to maintain independence of QA oversight
- Responsible for the implementing and communicating the QAM
- Maintains, approves, and implements the QAM

- Has joint signature authority, with the Laboratory Director and Technical Director for approval of quality documents, e.g., QAM, policies, and SOPs
- Directs controlled distribution of laboratory quality documents
- Provides Quality System training to all new personnel
- Reviews and approves documentation of analyst training records
- Serves as a focal point for QA and QC issues, reviews corrective actions and recommends resolution for recurring nonconformances within the laboratory
- Assists in maintaining regulatory analytical compliance, including maintaining certifications, and in this regard has signature authority for laboratory quality documents
- Monitors data quality measures via statistical methods to verify that the laboratory routinely meets stated quality goals
- Performs systems, data, contract compliance, and surveillance audits.
- Hosts external audits conducted by outside agencies
- Responsible for approving quality control reference data changes in the LIMS
- Oversees the selection, review, and approval of analytical subcontractors
- Prepares monthly QA Reports to management describing significant quality events
- Has the final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data
- Responsible for implementing internal/external audit findings corrective actions

4.2.15 Operations Manager/Laboratory Supervisor

- Reports directly to the Laboratory Director
- Supervises daily activities of the Operational Groups
- Schedules analytical operations
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Supervises maintenance of instruments and scheduling of repairs
- Works with the Project Managers and Group Leaders to assure the requirements of projects are met in a timely manner
- Supervises daily activities of the Sample Control Group.

4.2.16 Laboratory Technical Director

- Reports directly to Laboratory Director
- Responsible for the technical operation of the laboratory
- Has joint signature authority for QAM, policies, SOPs, and training records
- Performs technical training in area(s) of expertise
- Interfaces with management on technical needs and solving day-to-day technical issues
- Investigates technical issues related to projects as directed by QA
- Evaluates new methods, technical proposals, and statements of work
- Certifies technical laboratory personnel based on education and background to ensure that staff have demonstrated capability in the activities for which they are responsible by reviewing and signing analyst demonstrations.
- Performs other tasks as required by NELAC.

The Technical Director meets the requirements specified in Section 4.1.1.1 of NELAC standards.

4.2.17 Project Management Group Leader

- Reports directly to the Manager of Client Services
- Supervises daily activities of the Project Management and Administrative Groups
- Works with the Operations Manager and/or Group/Team Leaders to ensure the requirements of projects are met in a timely manner

4.2.18 Manager of Client Services

- Reports directly to Laboratory Director
- Supervises Field Analytical Services Group
- Supervises Service Centers
- Supervises daily activities of the customer
- Supervises Service Managers

4.2.19 Customer Service Managers (CSMs)

- Reports directly to the Manager of Client Services
- Defines customer requirements through project definition
- Assesses and assures customer satisfaction
- Provides feedback to management on changing customer needs
- Brings together resources necessary to ensure customer satisfaction.

4.2.20 Project Manager

- Reports directly to the Project Manager Group Leader
- Monitors analytical and QA project requirements for a specified project
- Acts as a liaison between the client and the laboratory staff
- Communicates project-specific requirements to all parties involved
- Assists the laboratory staff with interpretation of work plans, contracts, and QAPP requirements
- Oversees project data packages for completeness and compliance to client needs
- Has signature authority for final reports
- Keeps the laboratory and client informed of project status
- Together with the QA Manager, approves customer requested variances to methods and to standard laboratory protocols
- Monitors, reviews, and evaluates the progress and performance of projects
- Reports client inquiries involving data quality issues or data acceptability to the facility QA Manager and to the operations staff
- Conducts project reviews to assess the laboratory's performance in meeting customer requirements
- Prepares reissue requests for project data
- Responsible for meeting quality requirements.

4.2.21 Group Leader

- Reports directly to the Operations Manager
- Supervises daily activities of analyses within the group
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Evaluates instrument performance and supervises the calibration, preventive maintenance, and scheduling of repairs
- Oversees or performs review and approval of all analytical data
- Reports nonconformances to the appropriate managers
- Responsible for generation of SOPs for their section
- Responsible for meeting quality requirements.

4.2.22 Analyst

- Performs analytical methods and data recording in accordance with documented procedures
- Performs and documents calibration and preventive maintenance
- Performs data processing and data review procedures
- Reports nonconformances to the Supervisor/Manager and QA Manager
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures
- Responsible for meeting quality requirements defined in this LQM and other supporting QA procedures.

4.2.23 Sample Custodian

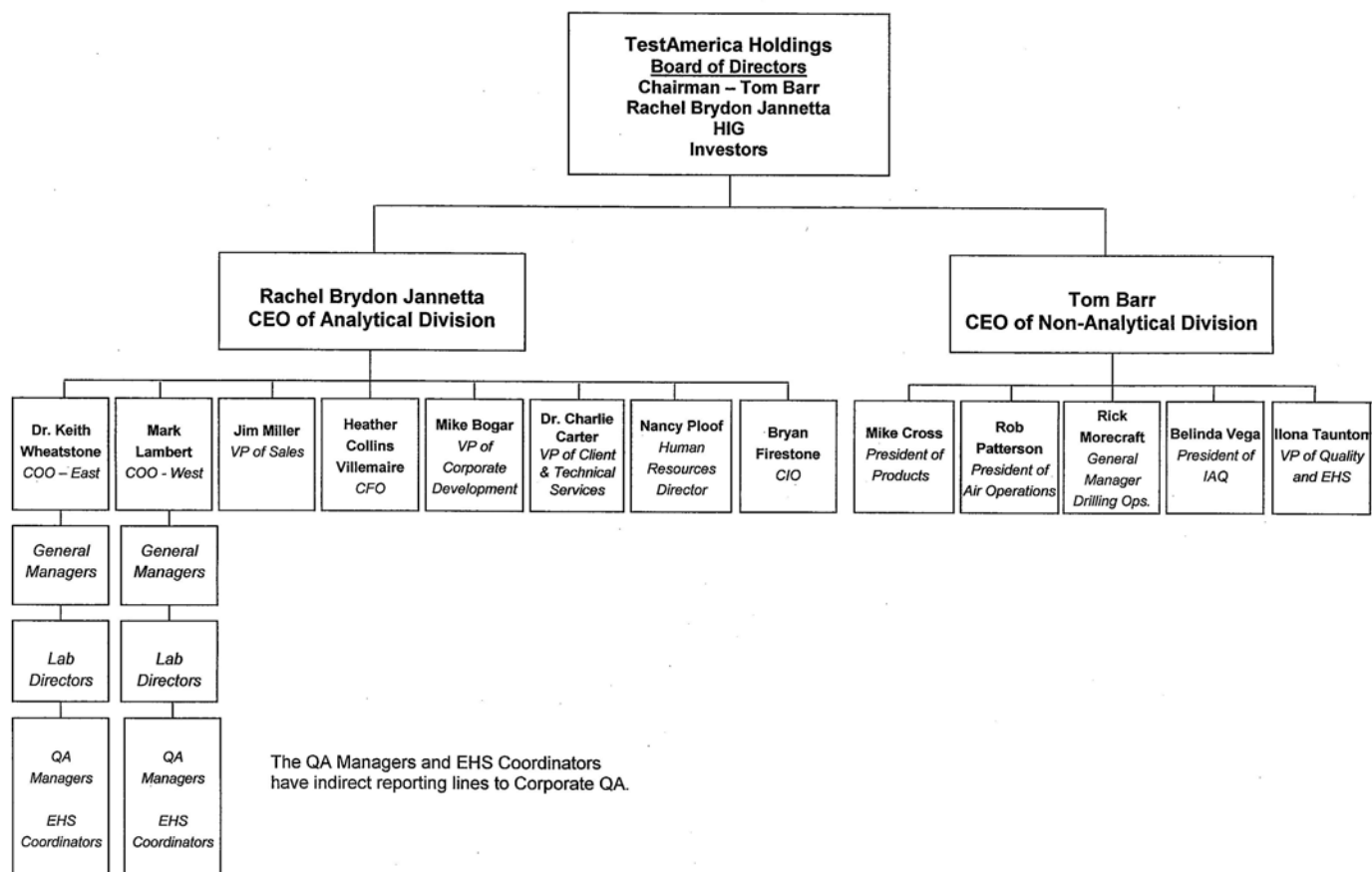
- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody
- Reports nonconformances associated with condition-upon-receipt of samples
- Logs samples into the LIMS
- Ensures that all samples are stored in the proper environment
- Assists Environmental Health and Safety staff with sample disposal
- Responsible for meeting quality requirements.

4.2.24 Report Production Staff

- Accurately generates and compiles analytical reports and associated deliverables for delivery to the client
- Responsible for meeting quality requirements
- Produce as needed reports that meet the NELAC requirements.

Figure 4-1.

Corporate Organization Chart



SECTION 5

QUALITY SYSTEM (NELAC 5.4.2)

5.1 QUALITY POLICY STATEMENT

The management of TestAmerica and TestAmerica North Canton are committed to providing data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols described in this manual.

In all aspects of the laboratory and business operations, management is dedicated in maintaining the highest ethical standards. An Ethics Policy sign-off can be viewed in Appendix 1. Training on ethical and legal responsibilities is provided annually and each employee signs off annually on the policy as a condition of employment.

It is TestAmerica's Policy to continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. The company recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.

TestAmerica North Canton strives to provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at TestAmerica North Canton plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The seven elements of the TestAmerica Ethics and Data Integrity Program include:

- An Ethics Policy (Policy CA-L-P-001) and Employee Ethics Statements (Appendix 1)
- An Ethics and Compliance Officer (ECO)
- A training program
- Self-governance through disciplinary action for violations
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct (SOP CA-L-S-001)
- Procedures and guidance for recalling data if necessary (SOP CA-L-S-001)
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16)

As an American Council of Independent Laboratories (ACIL) member, all TestAmerica laboratories adhere to the following ACIL Code of Ethics:

- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the ethical and quality standards of our industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION

The Laboratory Quality System is communicated through a variety of documents prepared by the laboratory and company management:

- Quality Assurance Manual (QAM) Template
- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system, e.g., checklists, preformatted bench sheets, forms.
- Laboratory SOPs – General and Technical
- Corporate TestAmerica QA/QC Policy Memorandums (refer to Section 3.4).
- Laboratory QA/QC Policy Memorandums (refer to Section 3.4).

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- TestAmerica QA/QC Policy Memorandum - Corporate
- Laboratory QA/QC Policy Memorandum
- Quality Assurance Manual
- Corporate SOPs and Policies

- Laboratory SOPs and Policies
- Other: Work Instructions (WI), memos, flow charts, etc.

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term “*analytical quality control*”. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples. The calculation of precision is described in Section 25.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery. The calculation of accuracy is described in Section 25.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories, and by the degree to which approval from the US EPA or other pertinent regulatory agencies is obtained for any procedure for which significant modifications have been made.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project) expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), inter-element corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc.

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory prepares a Control Limit Report that summarizes the precision and accuracy acceptability limits for analyses performed at TestAmerica North Canton. This summary includes an effective date, is updated each time new limits are generated, and is located in QC Browser. The charts are stored as hard copies. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, TestAmerica North Canton has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in Section 25.

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. TestAmerica North Canton routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Group Leader and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance Department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

In-house limits for all QC data must be evaluated and compared to the limits published in the methods for applicable matrices. Method limits will be employed until sufficient QC data are acquired. A minimum of 20 to 30 data points are recommended to establish the in-house QC limits. Calculated results of the QC (LCS) samples are evaluated by comparing against control limits (3-sigma).

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP or QAS.

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 17). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL (NELAC 5.4.3)

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled at each laboratory Facility:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

The Corporate staff posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These are collectively termed “Official Documents” and encompass the Policies and Procedures that all facilities are required to employ. These official documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving official documents is found in Corporate SOP CW-Q-S-001, Corporate Document Control and Archiving.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports. Discussion on records control is described in Section 15.

The maintenance of purchasing data is discussed in Section 9.

The maintenance of sales and marketing contracts is discussed in Section 7.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a control system for each document include a unique name and number, the number of pages of the item, the effective date, revision number, and the laboratory

name. The QA Department is responsible for the maintenance of the system, and maintains the items electronically on the Laboratory public drive or the QA Department files.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a staff member submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document, and retains the official document on file. The official document is provided as needed to those using it. Controlled documents shall be available at all locations where the operational activity described in the document is performed (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every 24 months, and revised as appropriate. For procedures associated with DoD project work, SOPs and Policies are reviewed every 12 months. Changes to documents occur when a procedural change warrants a revision of the document.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOPs NC-QA-002 and CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA/QC Department. Electronic copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, refer to SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP), and SOP NC-QA-027, Preparation and Management of Standard Operating Procedures.

Forms, worksheets, work instructions, and information are organized by department in the QA office. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP NC-QA-027.

6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 15.

SECTION 7

REVIEW OF WORK REQUEST

7.1 OVERVIEW

TestAmerica North Canton has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is TestAmerica's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and TestAmerica's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (Percent Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the lab's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval (refer to Section 8 for Subcontracting Procedures).

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and TestAmerica's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the

contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The review process is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the opportunity is forwarded to a Customer Service Manager (CSM) for review. The CSM contacts the appropriate Sales Executive (National Account Manager, Key Account Executive, Regional Account Executive, and/or Program Manager) to determine which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, reporting specifications, and available capacity to perform the work. The contract review process is outlined in SOP CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- Laboratory Customer Service Manager
- Laboratory Operations Manager
- Laboratory and/or Corporate Technical Director
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality Managers
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote, and makes final acceptance for their facility.
- Based on the level of discount extended for the project, approval of the General Manager or Sales Director may also be required.

The Customer Service Manager or local Account Executive then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her backup will fulfill the review requirements.

The Legal & Contracts Director (or their designee) maintains copies of all signed contracts. The Laboratory Director also maintains an electronic copy of any contract signed at the local level.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract will be distributed to and maintained by the Corporate Contracts Department and the applicable Account Executive. A copy of the contract will be filed electronically by the Laboratory Director. Quotes will be archived electronically in the laboratory quote module (TALs) or in the public shared drive if an off-TALs quote is submitted.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps email records or a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, TestAmerica assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the direct client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes, e.g., use of a non-standard method or modification of a method, and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory. Project-specific changes made after samples are in-house are communicated through Change Order forms.

Programmatic and/or method changes are communicated via email transmittal and/or in meetings with the applicable Operations Managers. If the modification includes use of a non-standard method, or significant modification of a method, documentation of the modification is made in the case narrative of the applicable data report(s).

TestAmerica strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

SECTION 8

SUBCONTRACTING OF TESTS (NELAC 5.4.5)

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the corporate network. The phrase “work sharing” refers to internal transfers of samples between company laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When we must outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the SOP on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

For DOD projects, the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-2. The subcontractor laboratory must receive project-specific approval from the DoD client before any samples are analyzed.

The QSM has five specific requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
2. Subcontractor laboratories must be approved by the specific DoD Component laboratory approval process.
3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.

4. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

Project Managers (PMs) or Customer Service Managers (CSM) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified network laboratory
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder)
- Firms listed as pre-qualified and currently under a subcontract with the company (in J.D.Edwards). A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation for the requested tests prior to sending samples.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses
- NELAC or A2LA-accredited laboratories
- In addition, the firm must hold the appropriate certification to perform the work required

All intra-company laboratories are pre-qualified for work sharing, provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to SOP CA-C-S-001, Work Sharing Process.

When the potential subcontract laboratory has not been previously approved, CSMs or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director/Manager. The Laboratory Director/Manager requests that the QA Manager begin the process of approving the subcontract

laboratory. The client must provide acknowledgement that the samples can be sent to that facility. (An e-mail is sufficient documentation; or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented.)

8.2.1 The QA Manager must ensure that the Subcontracting Approval Form (Figure 8-2) has been completed and have supporting documentation on file prior to initiation of any work. A letter or e-mail is sent to the lab requesting the following information:

8.2.1.1 If a lab is NELAC or A2LA-accredited:

8.2.1.1.1 Copy of necessary certifications verifying the required approvals are current. Ensure all needed analytes are included; some may not be accreditable (if so, document). Certificate and scope of international Standard accreditation are required, when applicable.

8.2.1.1.2 Insurance Certificate. This is required by the TestAmerica Chief Financial Officer

8.2.1.1.3 USDA soil permit, if available

8.2.1.2 For laboratories accredited by other agencies with an auditing program

8.2.1.2.1 Copy of necessary certifications verifying that the required approvals are current. Ensure all needed analytes are included; some may not be accreditable (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.

8.2.1.2.2 Insurance Certificate. This is required by the TestAmerica Chief Financial Officer

8.2.1.2.3 USDA soil permit, if available**

8.2.1.2.4 Description of Ethics and Data Integrity Plan

8.2.1.2.5 The most recent two sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action

8.2.1.2.6 State Audit with Corrective Action Response

8.2.1.2.7 Example final report to confirm format is compliant and provides the necessary information. (Minimally, it must be determined the Batch QC results are included in the laboratory reports, and data is appropriately qualified.)

8.2.1.2.8 A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA Manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review. (It may need to be sent elsewhere for evaluation.) This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously—minimum of six months—are

grandfathered.)

- 8.2.1.2.9** DoD work includes additional requirements as described in Section 8.1 above.
- 8.2.1.3** For laboratories performing tests that are unaccredited or accredited by an agency without an audit program:
 - 8.2.1.3.1** A copy of their Quality Assurance Manual (controlled, if possible). Ensure data quality limits for relevant methods are acceptable and that training procedures are adequate.
 - 8.2.1.3.2** A copy of necessary certifications (if available) verifying the required approvals are current. Ensure all needed analytes are included. Some may not be accreditable (if so, document). Certificate and scope of international Standard accreditation are required, when applicable.
 - 8.2.1.3.3** Insurance Certificate. This is required by the Test America Chief Financial Officer.
 - 8.2.1.3.4** USDA soil permit, if available**.
 - 8.2.1.3.5** Evidence of a current SOP per method. A copy of the first page and signature page of the SOP is acceptable. A Table of Contents including effective dates may also be acceptable. The SOP can be examined if an on-site audit is performed.
 - 8.2.1.3.6** Description of Ethics and Data Integrity Plan
 - 8.2.1.3.7** The most recent two sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.
 - 8.2.1.3.8** Example final report to confirm format is compliant and provides the necessary information. (Minimally, it must be determined the Batch QC results are included in the laboratory reports, and data is appropriately qualified.)
 - 8.2.1.3.9** Statement of Qualification (SOQ) or summary list of Technical Staff and Qualifications – position, education, and years of experience.
 - 8.2.1.3.10** DoD work includes additional requirements as described in Section 8.1 above.

**USDA permit is required if soils less than three feet deep from New York, North Carolina, South Carolina, Georgia, Florida, Tennessee, Alabama, Mississippi, Louisiana, Arkansas, Texas, Oklahoma, New Mexico, Arizona, California, Hawaii, or outside the continental U.S. are to be analyzed. These samples require special shipping measures (check with the EHS Department). It may be necessary to heat-treat the samples before shipping if the subcontract laboratory does not have a USDA permit; however, some analytes/tests may be irrelevant after heat treatment.

8.2.1.3.11 A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA Manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review. (It may need to be sent elsewhere for evaluation.) This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously—minimum of six months—are grandfathered.)

8.2.2 Once the information is received by the QA Manager, it is evaluated for acceptability and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site, along with the associated documentation and notify the Finance Group for J.D.Edwards.

8.2.3 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are to meet minimal standards. The company does not certify laboratories. The subcontractor is on our approved list, and can only be recommended to the extent that we would use them.

8.2.4 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contract Department. Any problems identified will be brought to Corporate QA attention.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor file on the intranet site. Complaints must be posted using the Vendor Performance Report (Form CW-F-WI-009).
- Information must be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all network laboratories and Corporate QA and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers, and Sales Directors.

8.3 OVERSIGHT AND REPORTING

The CSM or PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM or CSM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-3) and the form is retained in the project folder. For network laboratories, certifications can be viewed on the company website.

The Sample Control Department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within the network.

The PM will communicate with the subcontracted laboratory to monitor the status of the analyses, facilitate successful execution of the work and ensure the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory EDD, i.e., imported, the report must explicitly indicate the specific lab that produced the data and identify the specific methods and samples.

Note: The results submitted by a network work sharing laboratory may be transferred electronically and the results reported by the network work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director/Manager may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, Corporate QA must be informed, and the QA Manager will be required to verify adequacy of proficiency scores and certifications. The laboratory must also request a copy of the raw data to support the analytical results for the first project submitted to the subcontract laboratory unless the laboratory has NELAC accreditation. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. The QA Manager will request full documentation and qualify the subcontractor under the provisions above. The approval process should be completed within 30 calendar days of subcontracting.

Figure 8-1.
Example - Client-Approved Subcontractor Form

Client Information:

Client Name & Account Number: _____

Client Contact: _____

Client Address: _____

Project Information: (Please choose all applicable.)

❖ **Certification required:** ☐ **State** ☐ **NELAC** ☐ **A2LA** ☐ **Method**_____

☐ **Target compound**_____ ☐ **Other**_____ ☐ **N/A**

❖ **Required Turn around time (method provisional)**_____

Subcontractor's Information:

Subcontractor's Name: _____

Subcontractor's Contact: _____

Subcontractor's Email: _____

Subcontractor's Address: _____

Subcontractor's Phone Number: _____

Analytical Test/Compound/Method to be subcontracted: _____

Certification Statement:

I hereby give **[Insert Lab Name]** permission to use the above noted subcontractor for the above noted testing procedures/methods. I realize that the above subcontractor will be held liable for the validity of the above mentioned testing procedures/methods. All subcontractors shall meet the requirements as spelled out in project information and will follow all analytical holding times and turn around times for analytical reports. The subcontract laboratory, and not TestAmerica, will be held liable for liquidated damages for delays in subcontracted analytical reports and/or electronic data deliverables.

Client Signature

Date

Figure 8-2.

Example - Subcontracting Laboratory Approval Form (Initial / Renewal)

SUBCONTRACTING LABORATORY APPROVAL

Reference: Section 8 – Quality Assurance Manual

Date: _____
Laboratory: _____
Address: _____
Contact and e-mail address: _____
Phone: Direct _____ Fax _____

Requested Item ³	Date Received	Reviewed/ Accepted	Date
1. QA Manual ³			
2. Copy of State Certification ¹			
3. State Audit with Corrective Action Response (or NELAC or A2LA Audit) ³			
4. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response ^{1,3}			
5. SOQ or Summary list of Technical Staff and Qualifications ³			
6. SOPs for Methods to Be Loadshifted ^{2,3}			
7. USDA Soil Permit			
8. Insurance Certificate			
9. Sample Report ³			
10. For DoD Work: Statement that Lab quality system complies with QSM			
11. For DoD Work: Approved by specific DoD Component laboratory approval process			
11. Description of Ethics Program ³			

1 - Required when emergency procedures are implemented.

2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. This requirement may also be fulfilled by supplying a table of SOPs with effective dates.

3 – If the laboratory has NELAC accreditation, Item numbers 4 through 10 are not required.

On Site Audit Planned: YES NO If yes, Date Completed: _____ By Whom: _____

Comments: _____

Lab Acceptable for Subcontracting Work: YES NO Limitations: _____

QA Manager: _____ Date: _____
(Printed Name)

☐ Forwarded to Contract Coordinator by: _____ Date: _____

SECTION 9

PURCHASING SERVICES AND SUPPLIES (NELAC 5.4.6)

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Capital expenditures are made in accordance with the Controlled Purchases Procedure, CW-F-S-004. Only one quote is required where the item being purchased is a sole source product. Examples of sole source capital expenditures are laboratory test equipment, client specified purchases and building leases. A minimum of two quotes is required where the opportunity exists to source from more than one vendor. All documentation related to the purchase of capital items will be maintained in the individual CapEx files located in Corporate Purchasing. Data will be held in accordance with the record retention policy.

TestAmerica will enter into formal contracts with vendors when it is advantageous to do so. Contracts will be signed in accordance with the Authorization Matrix Policy, CW-F-P-002. Examples of items that are purchased through vendor contracts are laboratory instruments, consumables, copiers and office supplies. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

Non-capital expenditure items are purchased through the requisition and approval process in JD Edwards or through other TestAmerica authorized methods (approved web-sites, purchasing cards). Labs have the ability to select from the approved vendors in JD Edwards.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents must

meet with the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP CA-Q-S-001.

9.3.1 Purchasing

The nature of the analytical laboratory demands that all material used in any of the procedures is of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

9.3.2 Receiving

It is the responsibility of the Warehouse Manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to date the material when received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are kept on a backup disc located in the Wet Chemistry bullpen and online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

There are many different grades of analytical reagents available to the analyst. All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method.

- An expiration date cannot be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.
- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained in the Standard Logbook in each laboratory group.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a conductivity of less than 1 mmho/cm at 25°C. The conductivity is checked and recorded daily. If the water's conductivity is less than the specified limit, the Operations Manager must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Table 9-1 details specific storage instructions for reagents and chemicals. Section 22 discusses conditions for standard storage.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Operations Manager and/or the Laboratory Director/Manager. If they agree with the request the procedures outlined in Policy CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed, and Purchasing places the order.

Upon receipt of a new or used piece of equipment, it is given a short name, such as HP-20, added to the equipment list described in Section 21 that is maintained by the QA Department, and I.T. must be notified so it can be linked for backups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (see Section 20). For software, its operation must be deemed reliable and evidence of

instrument verification must be retained by the IT Department or QA Department as specified in the laboratory's procedure for software verification. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 21. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers or Operations Manager.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). The level of control used in the selection process is dependent on the anticipated spend and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (CW-F-WI-007 – refer to Figure 9-2).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Laboratory Director are consulted with vendor and product selection that have an impact on quality.

Table 9-1.
Storage of Reagents and Chemicals

Chemical	Storage Requirements
Concentrated Acids and Bases	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Bulk Dry Chemicals	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Working Solutions containing Organic Compounds	Stored as per method recommendation/ requirement. They are generally stored refrigerated at 4°C± 2°C.
Working Solutions containing only Inorganics	Stored at room temperature; refrigeration is optional.
Flammable Solvents	Stored in solvent cabinets at room temperature.
Non-Flammable Solvents	Stored separately from the flammable solvents in cabinets at room temperature.

Figure 9-2.
Example – JD Edwards Vendor Add Request Form



JD Edwards Vendor Add Request Form

Vendor name:	Lab location <u>and</u> individual making request:
Vendor address (remit to):	Vendor phone:
Vendor address (remit to):	Vendor fax:
Contact name:	Product / service provided:

Reason for Vendor Addition: Check all reasons that apply

<input type="checkbox"/> Cost Reduction	Estimated Annual Savings \$
<input type="checkbox"/> Replace Current Vendor	Reason?
	Vendor being Replaced?
<input type="checkbox"/> New Product / Service	Describe:
<input type="checkbox"/> ISO Approved (<u>Required for Aerotech / P&K only</u>)	

Small Business:

Does this vendor help us to meet our small business objectives: _____
If yes, which category: _____

Personal and Ethical Considerations:

Is there any personal conflict of interest with a TestAmerica employee and the vendor listed above? _____
Have ethical considerations been taken into account in your evaluation of this vendor? _____

Can this product be sourced from another TestAmerica facility? _____

Please complete form and email to NCPurchasing@testamericainc.com or fax to (330) 966-9275.

I approve the addition of this vendor:

Purchasing Manager - Patrick Eckman

Corporate Controller - Leslie Bowers

Form No. CW-F-WI-007

SECTION 10

SERVICE TO THE CLIENT (NELAC 5.4.7)

10.1 OVERVIEW

TestAmerica North Canton cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements discussed in Section 5. The laboratory has procedures to ensure confidentiality to clients (Sections 16 and 26).

Note: ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

10.2 SPECIAL SERVICES

The laboratory's standard procedures for reporting data are described in Section 26. When requested the following special services are provided:

- The laboratory will provide the client or the client's representative reasonable access to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- The laboratory will work with client-specified third party data validators as specified in the client's contract.
- The laboratory will provide the client with all requested information pertaining to the analysis of their samples. An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

10.3 CLIENT COMMUNICATION

Customer Service Managers (CSMs) and Project Managers (PMs) are an important communication link to the clients. The lab shall inform its clients of any delays in project completion as well as any non-conformances in either sample receipt (refer to Section 24) or sample analysis. Project Management will maintain ongoing client communication throughout the entire client project.

A Technical Director, Operation Manager, or Lab Supervisors are available to discuss any technical questions or concerns that the client may have.

10.4 REPORTING

The laboratory will work with the client to produce any special communication reports required by the contract.

10.5 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica Sales and Marketing teams periodically develop lab and client-specific surveys to assess client satisfaction.

SECTION 11

COMPLAINTS (NELAC 5.4.8)

11.1 OVERVIEW

TestAmerica North Canton believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that helps to continually improve processes and improving client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, communications, responsiveness, data, reports, invoicing and other functions expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for dealing with both external and internal complaints.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 13 (Corrective Actions) and is documented following SOPs CORP-QA-0010, Nonconformance and Corrective Action System, and S-C-002, Complaint Handling and Service Recovery. It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

11.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process and the documentation of the complaint.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving Complaints
- Complaint Investigation and Service Recovery

- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

11.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing, and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 13.

11.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 17)

SECTION 12

CONTROL OF NON-CONFORMING WORK (NELAC 5.4.9)

12.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. Refer to SOP CORP-QA-0010, Nonconformance and Corrective Action System, for details of the nonconformance process.

12.2 RESPONSIBILITIES AND AUTHORITIES

SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall, outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the company's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director/Manager, Operations Manager, Project Manager, or a member of the QA team may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures described in Section 13. This information may also need to be documented in logbooks and/or data review as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior laboratory management within 24 hours. The Senior Management staff is comprised of the Laboratory Director, QA Manager, Customer Service Manager, Operations Manager, I.T. Manager, H.R. Manager, and Technical Director. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO) and Quality Director within 24 hours.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director/Manager, QA Manager, ECOs, COOs (East and West), General Managers and the Quality Directors (East and West) have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

12.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

SOP CA-L-S-001 distinguishes between situations when it would be appropriate for the laboratory QA Manager and Laboratory Director/Manager (or his/her designee) to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting (Section 13) in lieu of the data recall determination form contained in SOP CA-L-S-001.

12.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system (Section 13).

On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

12.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 12.2, Paragraph 5 above.

Prior to suspension/restriction, confidentiality will be respected, and the problem and the required corrective and preventive action will be stated in writing and presented to the Laboratory Director/Manager.

The Laboratory Director/Manager shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not

be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 13 if one has not already been started. A copy of any meeting notes and agreed upon steps must be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the Internet. It is the responsibility of the Laboratory Director/Manager to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction, i.e., Project Management, Log-in, etc. Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Supervisor) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, Director of Client Services, and Director of Sales and Marketing should be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report as described in Section 13.

SECTION 13

CORRECTIVE ACTION (NELAC 5.4.10)

13.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Nonconformance Memos (NCM).

13.2 DEFINITIONS

- **Correction:** Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions are contained in the method-specific SOPs. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.
- **Corrective Action:** The action taken is not only a correction made to the immediate event, but a change in process, procedure or behavior that is required to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

13.3 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigation.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track Client complaints and provide resolution (see more on client complaints in Section 11).

13.3.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP

- QC outside of limits (non matrix related)
- Isolated Reporting / Calculation Errors
- Client Complaints

13.4 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

13.4.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 13-1 in SOP CORP-QA-0010, Nonconformance and Corrective Action System, provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Supervisor, Lab Director, QA Manager, or designee is consulted.

13.4.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM is used for this documentation.

13.4.3 Monitoring of the Corrective Actions

- The Department Manager/Supervisor and QA Manager is responsible to ensure that the corrective action taken was effective.
- Ineffective actions will be documented and re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM is entered into a database for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs for trends. Highlights are included in the QA monthly report (refer to Section 17). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.

- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

13.4.4 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements. (Section 16 includes additional information regarding internal audit procedures.)
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

13.5 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 12 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCM.

Table 13-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs.

Table 13-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, QAM Sections 20 and 21, and SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall. All corrective actions are reviewed at a minimum monthly by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

13.6 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out, and not erased, deleted, made illegible, or otherwise obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 13-1.
Example – General Corrective Action Procedures

* For Ohio VAP, method blank contamination must not exceed the RL if that analyte is a Contaminant of Concern for the project site.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Alkalinity	* Method Blank	310.1 2320B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	310.1 2320B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	310.1 2320B	<u>Total alkalinity:</u> 1 per batch of 20 samples	—	N/A
	Matrix Spike Duplicate	310.1 2320B	<u>Total alkalinity:</u> 1 per batch of 20 samples	—	N/A
	Duplicate	310.1 2320B	For carbonate, bicarbonate, hydroxide, alkalinity only. <u>Frequency:</u> 1 per batch of 10 samples <u>Criteria 310.1:</u> ≤ 20 % RPD ⁽³⁾ <u>Criteria 2320B:</u> ≤ 25 % RPD ⁽³⁾ <u>Corrective Action:</u> Flag data outside of limit.	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Ammonia	* Method Blank	350.1 350.2 SM4500 NH-E,F	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	350.1 350.2 SM4500 NH-E,F	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples	—	N/A
	Matrix Spike	350.1 350.2 SM4500 NH-E,F	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES¹	Method	RCRA (SW846)²
Ammonia (Cont'd)	Matrix Spike Duplicate	350.1 350.2 SM4500 NH-E,F	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	—	N/A
	Duplicate	350.1 350.2 SM4500 NH-E,F	N/A	—	N/A
Ammonia (TKN)	* Method Blank	351.2 351.3 SM4500 NO ₃	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Ammonia (TKN) (Cont'd)	Laboratory Control Sample	351.2 351.3 SM4500 NO ₃	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	351.2 351.3 SM4500 NO ₃	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	351.2 351.3 SM4500 NO ₃	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	351.2 351.3 SM4500 NO ₃	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
BOD	* Method Blank	405.1 SM5210B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	405.1 SM5210B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	405.1 SM5210B	N/A	—	N/A
	Matrix Spike Duplicate	405.1 SM5210B	N/A	—	N/A
	Duplicate	405.1 SM5210B	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES¹	Method	RCRA (SW846)²
Bromide	* Method Blank	300.0A ⁽⁵⁾	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample and Laboratory Control Sample Duplicate	300.0A ⁽⁵⁾	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples
	Matrix Spike	300.0A ⁽⁵⁾	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with MS outside of limit
	Matrix Spike Duplicate	300.0A ⁽⁵⁾	N/A	9056A	N/A
	Duplicate	300.0A ⁽⁵⁾	N/A	9056A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Chemical Oxygen Demand (COD)	* Method Blank	410.4 SM5220D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	410.4 SM5220D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	410.4 SM5220D	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	410.4 SM5220D	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	—	N/A
	Duplicate	410.4 SM5220D	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Chloride	* Method Blank	300.0A ⁵ 325.2 325.3	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Concentration must be less than the reporting limit <u>Corrective Action</u> : Rerun all samples associated with unacceptable blank	9056A	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Concentration must be less than the reporting limit <u>Corrective Action</u> : Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A ⁵ 325.2 325.3	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Corrective Action</u> : If not within control limits, rerun all associated samples	9056A	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Corrective Action</u> : If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A ^(b)	<u>Frequency</u> : 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Corrective Action</u> : Flag data outside of limit	9056A	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Methods 9251 Corrective Action</u> : If not within laboratory control limits, rerun all associated samples <u>Method 9056/9253</u> <u>Corrective Action</u> : Flag data associated with MS outside of limits

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Chloride (cont'd)	Matrix Spike Duplicate	325.2 325.3	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits/< 20 % RPD ⁽³⁾ <u>Corrective Action:</u> Flag data outside of limit <u>Method 9056:</u> MSD is not applicable
	Duplicate	300.0A ⁽⁵⁾ 325.2 325.3	N/A	9056A	N/A
Chlorine, Residual	* Method Blank	330.5	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	330.5	N/A	—	N/A
	Matrix Spike	330.5	N/A	—	N/A
	Matrix Spike Duplicate	330.5	N/A	—	N/A
	Duplicate	330.5	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> ≤ 20 % RPD ⁽³⁾ <u>Corrective Action:</u> Flag data outside of limit.	—	Water

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Chromium (Cr ⁺⁶)	* Method Blank	3500 Cr-D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	3500 Cr-D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples prepped <u>Criteria:</u> percent recovery for water must be within $\pm 15\%$ and for solids must be within $\pm 20\%$ <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	3500 Cr-D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit	3060A 7196A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Advisory limits are 75% - 125% recovery <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES¹	Method	RCRA (SW846)²
Chromium (Cr ⁺⁶) (Cont'd)	Matrix Spike Duplicate	3500 Cr-D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Advisory limits are 75% - 125% recovery <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	3500 Cr-D	N/A	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> ≤ 20 % RPD ⁽³⁾ limit <u>Corrective Action:</u> Flag data outside of limit.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Conductivity, Specific	* Method Blank	120.1 SM2510B	N/A	9050A	Not Applicable
	Laboratory Control Sample	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9050A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	120.1 SM2510B	N/A	9050A	N/A
	Matrix Spike Duplicate	120.1 SM2510B	N/A	9050A	N/A
	Duplicate	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: $\leq 20\%$ RPD ⁽³⁾ Corrective Action: Flag data outside of limit.	9050A	Frequency: 1 with each batch of samples processed not to exceed 10 samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Cyanide (Amenable)	* Method Blank	335.1 SM4500C N-G	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.1 SM4500C N-G	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.1 SM4500C N-G	N/A	9012A	N/A
	Matrix Spike Duplicate	335.1 SM4500C N-G	N/A	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within lab control limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	335.1 SM4500C N-G	N/A	9012A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Cyanide (Total)	* Method Blank	335.2 335.3 335.4 4500-CN E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.2 335.3 335.4 4500-CN E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.2 335.3 335.4 4500-CN E	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Cyanide (Total) (cont'd)	Matrix Spike Duplicate	335.2 335.3 335.4 4500-CN E	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	335.2 335.3 335.4	N/A	9012A	N/A
Flashpoint	* Method Blank	—	N/A	1010 ASTM D93-9	N/A
	Laboratory Control Sample	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike Duplicate	—	N/A	1010 ASTM D93-9	N/A
	Duplicate	—	<u>Frequency:</u> 1 per batch of ≤20 samples per matrix <u>Criteria:</u> RPD ⁽³⁾ must be ≤ 20% <u>Corrective Action:</u> Flag data associated with unacceptable Duplicate	1010 ASTM D93-9	<u>Frequency:</u> 1 per batch of ≤20 samples per matrix <u>Criteria:</u> RPD ⁽³⁾ must be ≤ 20% <u>Corrective Action:</u> Flag data associated with unacceptable Duplicate

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Fluoride	* Method Blank	300.0A ⁽⁵⁾ 340.2 SM4500F-C,ISE	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable
	Laboratory Control Sample	300.0A ⁽⁵⁾ 340.2 SM4500F-C,ISE	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples
	Laboratory Control Sample Duplicate	300.0A ⁽⁵⁾ SM4500F-C,ISE	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Flouride (Cont'd)	Matrix Spike	300.0A ⁽⁵⁾ 340.2 SM4500F-C,ISE	Frequency: 1 per 10 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with outside of limit
	Matrix Spike Duplicate	340.2 SM4500F-C,ISE	Frequency: 1 per 20 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	340.2	N/A
	Duplicate	300.0A ⁽⁵⁾ 340.2 SM4500F-C,ISE	N/A	9056A	Frequency: 1 with each batch of samples processed Criteria: RPD ⁽³⁾ must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD ⁽³⁾ limits

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Hardness	* Method Blank	130.2 2340B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	130.2 2340B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	130.2 2340B	<u>Method 130.2:</u> 1 per 20 samples <u>Method 2340B:</u> <u>Frequency , Criteria, and Corrective Action:</u> See ICP Metals Method 200.7 Requirements	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Hardness (cont'd)	Matrix Spike Duplicate	130.2 2340B	<u>Method 130.2:</u> 1 per 20 samples <u>Method 2340B:</u> <u>Frequency, Criteria, and Corrective Action:</u> See ICP Metals Method 200.7 Requirements	—	N/A
	Duplicate	130.2 2340B	N/A	—	N/A
Iron, Ferrous & Ferric	* Method Blank	3500-Fe D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	3500-Fe D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Iron, Ferrous & Ferric (Cont'd)	Matrix Spike	3500-Fe D	<u>Frequency</u> : 1 every 10 samples <u>Criteria</u> : Must be within laboratory QC limits <u>Corrective Action</u> : Flag associated data outside of limit	—	N/A
	Matrix Spike Duplicate	3500-Fe D	<u>Frequency</u> : 1 every 10 samples <u>Criteria</u> : Must be within laboratory QC limits <u>Corrective Action</u> : Flag associated data outside of limit	—	N/A
	Duplicate	3500-Fe D	N/A	—	N/A
Nitrate	* Method Blank	300.0A ⁽⁵⁾ 353.2 SM4500-NO ₃ -E	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Concentration must be less than the reporting limit <u>Corrective Action</u> : Rerun all samples associated with unacceptable blank	9056A	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Concentration must be less than the reporting limit

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Nitrate (Cont'd)	Laboratory Control Sample	300.0A ⁽⁵⁾ 353.2 SM4500-NO ₃ -E	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A ⁽⁵⁾ 353.2 SM4500-NO ₃ -E	Frequency: 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A ⁽⁵⁾ 353.2 SM4500-NO ₃ -E	Frequency: 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Nitrate (cont'd)	Duplicate	300.0A ⁽⁵⁾ 353.2 SM4500- NO ₃ -E	N/A	9056A	<u>Frequency:</u> 1 per 10 samples <u>Criteria:</u> RPD ⁽³⁾ must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples
Nitrite	* Method Blank	300.0A ⁽⁵⁾ 353.2	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A ⁽⁵⁾ 354.1 353.2	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Nitrite (Cont'd)	Matrix Spike	300.0A ⁽⁵⁾ 354.1 353.2	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A ⁽⁵⁾ 354.1 353.2	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	N/A
	Duplicate	300.0A ⁽⁵⁾ 354.1 353.2	N/A	9056A	<u>Frequency:</u> 1 per 10 samples <u>Criteria:</u> RPD ⁽³⁾ must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Nitrate-Nitrite	* Method Blank	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	353.2	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
pH	* Method Blank	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Laboratory Control Sample	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9040B 9045C	N/A
	Matrix Spike	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Matrix Spike Duplicate	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Duplicate	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 10 samples per matrix Criteria: $\leq 20\%$ RPD ⁽³⁾ limit Corrective Action: Flag data outside of limit.	9040B 9045C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are $\leq 20\%$ RPD ⁽³⁾ Corrective Action: Flag data associated with unacceptable Duplicate
Phenolics	* Method Blank	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Phenolics (Cont'd)	Laboratory Control Sample	420.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9065	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	420.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable matrix spike	9065	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data
	Matrix Spike Duplicate	420.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data	9065	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Phosphorus (Total and Ortho-phosphate)	* Method Blank	300.0 ^(4,5) 365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0 ^(4,5) 365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0 ^(4,5) 365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 per 10 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data associated with MS outside of limits
	Matrix Spike Duplicate	365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 per 20 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit	9056A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Phosphorus (Total and Ortho-phosphate) (Cont'd)	Duplicate	300.0 ^(4,5) 365.3 365.1 SM4500P-E	N/A	9056A	<u>Frequency</u> : 1 with each batch of samples processed <u>Criteria</u> : RPD ⁽³⁾ must be within laboratory control limits <u>Corrective Action</u> : Flag data associated with duplicates outside of laboratory RPD ⁽³⁾ limits
Solids	* Method Blank	160.1 160.2 160.3 160.4 160.5 SM2540E	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Concentration must be less than the reporting limit <u>Corrective Action</u> : If analyte level in method blank is \geq RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed.	—	N/A
	Laboratory Control Sample	160.1 160.2 160.3 160.4 160.5 SM2540E	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Corrective Action</u> : If not within laboratory control limits, reprepare and rerun all associated samples	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Solids (Cont'd)	Matrix Spike	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Matrix Spike Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples <u>Criteria:</u> Sample results should agree within 20% if both the sample and sample duplicate results are > 5 X RL <u>Corrective Action:</u> Flag data outside of limit	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Sulfate	* Method Blank	300.0A ⁽⁵⁾ 375.4	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9038 9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A ⁽⁵⁾ 375.4	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9038 9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Method 9038 Criteria:</u> Percent recovery must be within $\pm 15\%$ <u>Method 9056 Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS (ICV)

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Sulfate (cont'd)	Matrix Spike	300.0A ⁽⁵⁾ 375.4	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9038 9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples (9038) or 20 samples (9056) <u>Method 9038 Criteria:</u> Limits are 75% - 125% recovery <u>Method 9056 Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	300.0A ⁽⁵⁾ 375.4	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9038 9056A	N/A
	Duplicate	300.0A ⁽⁵⁾ 375.4	N/A	9038 9056A	N/A
Sulfide	* Method Blank	376.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9030A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Sulfide (Cont'd)	Laboratory Control Sample	376.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9030A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data
	Matrix Spike	376.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9030A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data
	Matrix Spike Duplicate	376.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9030A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data Method 9034: Not Applicable
	Duplicate	376.1	N/A	9030A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Total Organic Carbon (TOC)	* Method Blank	415.1 SM5310D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	415.1 SM5310D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	415.1 SM5310D	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Total Organic Carbon (TOC) (cont'd)	Matrix Spike Duplicate	415.1 SM5310D	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike Duplicate
	Duplicate	415.1 SM5310D	N/A	9060 Walkley-Black	Not Applicable
Total Organic Halides (TOX)	* Method Blank	450.1 ⁽⁵⁾	<u>Frequency:</u> 1 with each set of 8 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9020B	<u>Frequency:</u> Run in duplicate between each group of 8 analytical determinations <u>Criteria:</u> Concentration less than reporting limit or less than 2 X MDL or RL whichever is lower <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	450.1 ⁽⁵⁾	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery of analyte must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS (ICV)	9020B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery of analyte must be within 90-110% <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS (ICV)

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Total Organic Halides (TOX) (cont'd)	Matrix Spike	450.1 ⁽⁵⁾	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	<u>Frequency:</u> 1 per batch of 10 samples <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP NO. CORP-WC-0001
	Matrix Spike Duplicate	450.1 ⁽⁵⁾	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	N/A
	Duplicate	450.1 ⁽⁵⁾	N/A	9020B	N/A
Turbidity	* Method Blank	180.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Turbidity (cont'd)	Laboratory Control Sample	180.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	180.1	N/A	—	N/A
	Matrix Spike Duplicate	180.1	N/A	—	N/A
	Duplicate	180.1	<u>Frequency:</u> 1 per 10 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit Not Applicable.	—	N/A
Mercury by CVAA & CVAFS	* Method Blank	200 series 1631E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0003. Exception: If blank is above RL, and samples are ND.	7000A series	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0003. Exception: If blank is above RL and samples are ND.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Mercury by CVAA & CVAFS (Cont'd)	Laboratory Control Sample	200 series 1631E	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery of analyte must be within ± 20 %. 1631E is $\pm 23\%$</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0003. Exception: If samples are ND, results are reported.</p>	7000A series	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery of analyte must be within ± 20 %</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0003. Exception: If samples are ND, results are reported.</p>
	Matrix Spike	200 series 1631E	<p><u>Frequency:</u> with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125%</p> <p><u>Criteria:</u> Recovery should be within 75-125 %</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable MS. (See SOP CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)</p>	7000A series	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Recovery should be within 75-125 %</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable MS. (See SOP CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)</p>
	Matrix Spike Duplicate	200 series 1631E	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% RPD 24%</p> <p><u>Criteria:</u> Recovery should be within 75-125 % , RPD⁽³⁾ should be within 20%</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable MSD SOP CORP-MT-0003</p>	7000A series	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Recovery should be within 75-125 % , RPD⁽³⁾ should be within 20%</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable MSD SOP CORP-MT-0003</p>

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Mercury by CVAA & CVAFS (Cont'd)	Duplicate	200 series 1631E	N/A	7000A series	N/A
ICP Metals	* Method Blank	200.7 200.8	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid.</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0001</p>	6010B 6020	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid.</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0001</p>
	Laboratory Control Sample	200.7 200.8	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery of analyte must be \pm 85-115%. If LCS is biased high and samples are <RL, the results are valid.</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0001</p>	6010B 6020	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery of analyte must be \pm 20 %. If LCS is biased high and samples are <RL, the results are valid.</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0001. If samples are ND, results are reported.</p>

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
ICP Metals (Cont'd)	Matrix Spike	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001
	Matrix Spike Duplicate	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125%, RPD ⁽³⁾ must be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125%, RPD ⁽³⁾ must be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001
	Duplicate	200.7 200.8	Not Applicable	6010B 6020	Not Applicable
	Serial Dilution	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> 10% difference. 10% difference only applied if sample results are >50 times IDL. <u>Corrective Action:</u> Flag data associated with unacceptable Serial Dilution SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> 10% difference. 10% difference only applied if sample results are >50 times IDL. <u>Corrective Action:</u> Flag data associated with unacceptable Serial Dilution SOP CORP-MT-0001

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Footnotes

- ¹ National Pollutant Discharge Elimination System
- ² Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- ³ RPD-Relative Percent Difference
- ⁴ Orthophosphate only
- ⁵ Method not listed in 40 CFR Part 136. Method 300.0 is a proposed 40CFR method. Specific state and/or region approval is required for NPDES.
- ⁶ Current promulgated method is a Guidance Method Only, SW-846, Final Update III, Rev.3, 12/96.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Aromatic Volatiles by GC	* Method Blank	602	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	602	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery must be within acceptance limits given in method for each analyte <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	602	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Aromatic Volatiles by GC (Cont'd)	Matrix Spike Duplicate	602	N/A	8021B	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action</u> : Flag data associated with unacceptable Matrix Spike
	Duplicate	602	N/A	8021B	N/A
	Surrogates	602	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : Reprep and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	602	Optional: Internal standards are added to the method blank and all samples (QC included).	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Halogenated Volatiles by GC	* Method Blank	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Halogenated Volatiles by GC (Cont'd)	Duplicate	--	N/A	8021B	N/A
	Surrogates	--	N/A	8021B	<p>Surrogates spiked into method blank and all samples (QC included)</p> <p><u>Method Blank Criteria and LCS:</u> All surrogates must be within laboratory established control limits before sample analysis may proceed.</p> <p><u>Sample Criteria:</u> Reprep and reanalyze samples or flag sample data not meeting surrogate criteria.</p>
	Internal Standards	--	N/A	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Herbicides	Laboratory Control Sample	615 ³	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within acceptance limits given in method for each analyte <u>Corrective Action:</u> Re-extract all samples associated with unacceptable LCS	8151A	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike	615 ³	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> Percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8151A	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	615 ³	N/A	8151A	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable matrix spike sample

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Herbicides (cont'd)	Duplicate	615 ³	N/A	8151A	N/A
	Surrogates	615 ³	N/A	8151A	<p>Surrogates spiked into method blank and all samples (QC included)</p> <p><u>Method Blank Criteria and LCS:</u> All surrogates must fall within laboratory established control limits before sample analysis may proceed.</p> <p><u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria</p>
	Internal Standards	615 ³	N/A	8151A	Optional
Pesticides/ PCBs	* Method Blank	608	<p><u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>	8081A 8082	<p><u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit</p> <p><u>Corrective Action:</u> Reprepare and reanalyze all samples associated with unacceptable blank</p>

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Pesticides/ PCBs (Cont'd)	Laboratory Control Sample	608	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery must be within acceptance limits given in method for each analyte <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	8081A 8082	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	608	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8081A 8082	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Pesticides/ PCBs (cont'd)	Matrix Spike Duplicate	608	N/A	8081A 80882	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	608	N/A	8081A 8082	N/A
	Surrogates	608	Not specified in method	8081A 8082	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS:</u> Results must fall within laboratory established control limits <u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
Petroleum Hydro- carbons	* Method Blank	1664A	<u>Frequency:</u> 1 with each preparation batch <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9071B	<u>Frequency:</u> 1 with each preparation batch <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Petroleum Hydrocarbons (Cont'd)	Laboratory Control Sample	1664A	<u>Frequency:</u> 1 with each analytical batch <u>Criteria:</u> Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	9071B	<u>Frequency:</u> 1 with each analytical batch <u>Criteria:</u> Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	1664A	<u>Frequency:</u> 1 with every 10 samples per site <u>Criteria:</u> See percent recovery limits in SOP, NC-WC-0084 <u>Corrective Action:</u> See SOP, NC-WC-0084	9071B	<u>Frequency:</u> 1 with every 10 samples per site <u>Criteria:</u> See percent recovery limits in SOP, NC-WC-0084 <u>Corrective Action:</u> See SOP, NC-WC-0084
	Matrix Spike Duplicate	1664A	<u>Frequency:</u> 1 with every 20 samples per site <u>Criteria:</u> See percent recovery and RPD limits in SOP, NC-WC-0084 <u>Corrective Action:</u> See NC-WC-0084	9071B	<u>Frequency:</u> 1 with every 10 samples per site <u>Criteria:</u> See percent recovery and RPD limits in SOP, NC-WC-0084 <u>Corrective Action:</u> See NC-WC-0084
	Duplicate	1664A	N/A	9071B	N/A

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Purgeable Halocarbons by GC	* Method Blank	601	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	8021B	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	601	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery must be within acceptance limits given in method for each analyte <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	8021B	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	601	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8021B	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Purgeable Halocarbons by GC (Cont'd)	Matrix Spike Duplicate	601	N/A	8021B	<u>Frequency</u> : 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria</u> : percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action</u> : Flag data associated with unacceptable Matrix Spike
	Duplicate	601	N/A	8021B	N/A
	Surrogates	601	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	601	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Semivolatiles	Matrix Spike	625	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8270C	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	625	N/A	8270C	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Semivolatiles (Cont'd)	Duplicate	625	N/A	8270C	N/A
	Surrogates	625	<p>Surrogates spiked into method blank and all samples (QC included)</p> <p><u>Method Blank and LCS Criteria:</u> All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery.</p> <p><u>Sample Criteria:</u> Re-extract samples or flag sample data not meeting surrogate criteria</p>	8270C	<p>Surrogates spiked into method blank and all samples (QC included)</p> <p><u>Method Blank and LCS Criteria:</u> All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery.</p> <p><u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria</p>
	Internal Standards	625	<p><u>Frequency:</u> Internal standards spiked into method blank and all samples (QC included)</p> <p><u>Criteria:</u> All internal standard recoveries must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag sample data not meeting internal standard recovery requirements</p>	8270C	<p>Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard.</p> <p>The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.</p>

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Volatiles by GC/MS	* Method Blank	624	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	624	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Volatiles by GC/MS (Cont'd)	Matrix Spike	624	<u>Frequency:</u> 1 per ≤ 20 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	624	N/A	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	624	N/A	8260B	N/A

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Volatiles by GC/MS (cont'd)	Surrogates	624	<p>Surrogates spiked into Method Blank and all samples (QC included)</p> <p><u>Method Blank Criteria:</u> All surrogates must be in control before sample analysis may proceed.</p> <p><u>Sample Criteria:</u> Re-extract samples or flag sample data not meeting surrogate criteria</p>	8260B	<p>Surrogates spiked into Method Blank and all samples (QC included)</p> <p><u>Method Blank Criteria and LCS:</u> All surrogates must be in control before sample analysis may proceed.</p> <p><u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria</p>
	Internal Standards	624	<p><u>Frequency:</u> Internal standards spiked into method blank and all samples (QC included)</p> <p><u>Criteria:</u> All internal standard recoveries must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag sample data not meeting internal standard recovery requirements</p>	8260B	<p>Internal Standards are added to all samples (QC samples included).</p> <p>Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard.</p> <p>The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.</p>

Footnotes

- ¹ National Pollutant Discharge Elimination System
- ² Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996)
- ³ Method not listed in 40 CFR Part 136

SECTION 14.0

PREVENTIVE ACTION (NELAC 5.4.11)

14.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes our TestAmerica North Canton commitment to the Quality Assurance (QA) program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc.

The monthly Quality Assurance Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, Ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's Corrective Action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

14.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, management review, and the Management of Change process (see below).

Note: There may be varying levels of formality and documentation during the preventive action process due to the simplicity/complexity of the action taken.

14.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 17). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

14.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP CA-Q-S-003, Management of Change.

SECTION 15.0

CONTROL OF RECORDS (NELAC 5.4.12)

TestAmerica North Canton maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued as described in SOP NC-QA-0019, Records Information Management.

15.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 15-1. Quality records are maintained by the Quality Assurance (QA) Manager in a database which is backed up as part of the regular network backup. Records are of two types--either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the Records Manager.

Table 15-1. Record and Retention Schedule

Type of Record	Retention	Disposition
General Laboratory Documents		
Instrument output	5 yrs from project completion	Shred or burn
Quality control data	5 yrs from project completion	Shred or burn
Field sample data	5 yrs from project completion	Shred or burn
Final analytical reports	5 yrs from project completion	Shred or burn
Instrument logbooks	5 yrs from last entry	Shred or burn
Equipment monitoring & maintenance records	5 yrs from last entry	Shred or burn
Instrument calibration records	5 yrs from last entry	Shred or burn
Standard preparation logs	5 yrs from last entry	Shred or burn
Standards certificates	5 yrs from last entry	Shred or burn
Measurement & test equipment logs (e.g., refrig., balances, etc.)	5 yrs from last entry	Shred or burn
Method & instrument validation records	5 yrs from last entry	Shred or burn
Instrument manuals	Retain until superseded	Trash
Project management files	5 yrs from date of archival	Shred or burn
Quotes & proposals	2 yrs from date of expiration	Shred or burn

Type of Record	Retention	Disposition
LQM, policies, & SOPs	5 yrs from date of archiving	Shred or burn
Analyst demonstrations of proficiency	5 yrs from date of archival	Shred or burn
Quality assurance audits	5 yrs from last entry	Shred or burn
Certifications & approvals	5 yrs from last entry	Shred or burn
Employee signature list	5 yrs from date of archival	Shred or burn
MDL Studies	5 yrs from last entry	Shred or burn
Performance testing studies	5 yrs from last entry	Shred or burn
QA reports to management	5 yrs from last entry	Shred or burn
Quality control charts	5 yrs from last entry	Shred or burn
Environment, Health and Safety Records		
Medical records	Retain while active & 30 yrs from last entry	Shred or burn
Employee exposure & monitoring records	Retain while active & 30 yrs from last entry	Shred or burn
Workers compensation files & first report of injury	Retain while active & 30 yrs from last entry	Shred or burn
Accident logs (OSHA Form 200)	5 yrs from last entry	Shred or burn
Accident reports	5 yrs from last entry	Shred or burn
Environmental permits	5 yrs from last entry	Shred or burn
Environmental management, e.g., discharge reports	5 yrs from last entry	Shred or burn
Health & safety audits	5 yrs from last entry	Shred or burn
Chemical Hygiene Plan	5yrs from archival	
Safety Inspections	5 yrs from last entry	Shred or burn
TLD exposure records	5 yrs from last entry	Shred or burn
EH&S training	5 yrs from last entry	Shred or burn
Accounting	See Accounting and Controls Procedures Manual	
Administrative		
Personnel records (not including medical or disability records)	7 years from last entry	Shred or burn

All records are legible and stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. Records are maintained for a minimum of five years unless other wise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.3. Policy CW-L-P-001, Record Retention, provides additional information on record retention requirements.

15.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 15-3 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 15-2. Special Record Retention Requirements

Program	Retention Requirement
Ohio – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
OSHA - 40 CFR Part 1910	30 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement and others as negotiated.
Ohio Voluntary Action Program	10 years

15.1.2 All records are held secure and in confidence. Records maintained at the laboratory are located in the Records Storage area of the Warehouse. Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Logs are maintained in each storage box to note removal and return of records.

15.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is

maintained as hardcopy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, see Section 20.12.1, Computer and Electronic Data Related Requirements, for more information.

15.1.4 The record-keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. (Records stored off site should be accessible within two days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory copy of the Chain-of-Custody is stored with the invoice and the Work Order sheet generated by LIMS. The Chain-of-Custody would indicate the name of the sampler. If any sampling notes are provided with a Work Order, they are kept with this package.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record-keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files, set format for what is included with a given analytical data set. SOP NC-QA-0019, Records Information Management, outlines this procedure. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "Sampled by," "Prepared by," "Reviewed by", or "Analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost, and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy which was scanned.
- Also refer to Section 20.13.1, "Computer and Electronic Data Related Requirements".

15.2 TECHNICAL AND ANALYTICAL RECORDS

15.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for sample preparation, performance of each analysis and checking of results.

15.2.2 Observations, data and calculations are recorded at the time they are made and are identifiable to the specific task.

15.2.3 Changes to hardcopy records shall follow the procedures outlined in Sections 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include (previous discussions relate where most of this information is maintained – specifics may be added below):

- Laboratory sample ID code
- Date of analysis and time of analysis is required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available. Instrument logs may be in electronic format.
- analysis type
- all manual calculations and manual integrations
- analyst or operator initials/signature
- sample preparation
- test results
- standard and reagent origin, receipt, preparation, and use
- calibration criteria, frequency and acceptance criteria
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions
- quality control protocols and assessment
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

15.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

15.3.1 Sample Handling Records

Sample handling and tracking is discussed in Section 24. Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

15.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hardcopy form (see Table 15-1).

15.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

15.5.1 All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure, and in confidence to the client. Certification-related records are available to the accrediting body upon request.

15.5.2 All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

15.5.3 Records that are stored or generated by computers or personal computers have hardcopy, write-protected backup copies, or an electronic audit trail controlling access.

15.5.4 TestAmerica North Canton has a record management system for control of instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting. Benchsheets are filed sequentially per method.

15.5.5 Records are considered archived when moved to Records Storage. Access to archived hard-copy information is documented with an access log and in/out records is used in archived boxes to note data that is removed and returned. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to the data is limited to laboratory and company employees.

15.5.6 In the event that the laboratory transfers ownership or goes out of business, TestAmerica North Canton shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous five years of such action.

15.5.7 Records Disposal

15.5.7.1 Records are removed from the archive and disposed after five years, unless otherwise specified by a client or regulatory requirement. On a project-specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration.

15.5.7.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

15.5.7.3 If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required. Refer to Policy CW-L-P-001, Records Retention.

SECTION 16

AUDITS (NELAC 5.4.13)

16.1 OVERVIEW

Audits measure laboratory performance and insure compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the quality of results produced by the laboratory, including how well the policies and procedures of the QA system and the Ethics and Data Integrity Program are being executed. They are also instrumental in identifying areas where improvement in the QA system will increase the reliability of data. There are two principle types of audits: Internal and External. Internal audits are performed by laboratory or corporate personnel. External audits are conducted by regulators, clients or third-party auditing firms. In either case, the assessment to program requirements is the focus.

Table 16-1. Audit Types and Frequency

Internal Audits	Description	Performed by	Frequency
	Analyst & Method Compliance	QA Department or Designee	- 100% of all methods over a two year period. - 100% of all analysts annually.
	Instrument	QA Department or Designee	100% of all organic instruments and any inorganic chromatography instruments. Annually.
	Work Order/ Final Report	QA Department or Designee	- 1 complete report each month.
	Support Systems	QA Department or Designee	- Annual for entire labs support departments & equipment (e.g., thermometers, balances), can be divided into sub-sections over the course of the year.
	Performance Audits (Double-Blind PTs)	Corporate QA, Laboratory QA Department or Designee	- As needed.
	Special	QA Department or Designee	- As Needed
External Audits	Description	Performed by	Frequency
	Program / Method Compliance	Regulatory Agencies, Clients, accreditation organizations	- As required by program and/or clients needs
	Performance Audits	Provided by a third party.	- As required by a client or regulatory agency. Generally provided semi-annually through the analysis of PT samples.

16.2 INTERNAL AUDITS

Annually, the laboratory prepares a schedule of internal audits to be performed throughout the year. As previously stated, these audits verify and monitor that operations continue to comply with the requirements of the laboratory's QA Manual and the Corporate Ethics Program. A

schedule of internal audits is maintained by the QA Manager in the *Internal Audit Workbook*. An example can be found in Figure 16-1.

It is the responsibility of the QA Manager to plan and organize audits in consideration of the laboratory workload and the department personnel schedules so that all pertinent personnel and operations are thoroughly reviewed. When designees (other than QA Department personnel and approved by the QA Manager) perform audits, the QA Manager shall ensure that these persons do not audit their own activities, except when it can be demonstrated that an effective audit will be carried out. In general, the auditor:

- Is neither the person responsible for the process being audited nor the immediate supervisor of the person responsible for the project/process.
- Is free of any conflicts of interest.
- Is free from bias and influences that could affect objectivity.

Laboratory personnel (e.g., supervisors and analysts) may assist with both method and support system audits as long as the items listed in the above paragraph are observed. These audits are conducted according to defined criteria listed in the checklists of the *Internal Audit Workbook*. These personnel must be approved by the QA Manager; and must complete the audit checklists in their entirety. This process introduces analyst experience and insight into the laboratory's auditing program.

The auditor must review the previous audit report and identify all items for verification of corrective actions. A primary focus will be dedicated to the ability of the laboratory to correct root-cause deficiencies and that the corrective action has been implemented and sustained as documented.

16.2.1 Systems

An annual Systems audit is required to ensure compliance to analytical methods and SOPs, laboratory Data Integrity and Ethics Policies, NELAC quality systems, client, and State requirements. This audit is performed in portions throughout the year through method, analyst, instrument, Work Order/Final Report, and support system audits. Audits are documented and reported to management within one week of their performance. Systems audits cover all departments of the facility--both operational and support. The multiple audits are compiled into one systems audit package at the end of the year (*Internal Audit Workbook*).

16.2.1.1 Method, Analyst, Instrument and Work Order/Final Report Audits

Procedures for the method compliance, analyst, instrument and work order/final report audits are incorporated by reference to SOP CA-Q-S-004, Method Compliance and Data Authenticity Audits. These audits are not mutually exclusive. For example, the performance of a method audit will also cover multiple analysts and instruments. The laboratory's goal is to annually review all analysts and instruments as described in SOP CA-Q-S-004. The laboratory will also audit all methods within a two-year time period, and audit a minimum of one Work Order/Final Report from receiving through reporting on a monthly basis.

16.2.1.2 Support Systems

Support system audits are performed to ensure that all departments & ancillary equipment are operating according to prescribed criteria. Support system audits include the review of both non-analytical and operational departments. Support equipment audits, e.g., metrology items, include the review of balance calibrations, weight calibrations; water quality testing, etc.. Non-analytical may include sample receiving and bottle preparation. These types of support audits ensure that the operations are being performed to support ethical data as well as ensuring the accuracy & precision of the utilized equipment.

These audits can be performed in portions throughout the year or in one scheduled session. However, the audit schedule must document that these aspects are reviewed annually. Many of the metrology systems are considered to be surveillance activities that can be monitored by QA personnel or delegated to specified department personnel. These surveillance activities are performed on a semi-annual basis unless issues warrant a greater frequency or previous audits continually showing no deficiencies allow the frequency to be reduced to once a year.

An example audit checklist can be found in Figure 16-2. Instructions for reporting findings are included in the *Internal Audit Workbook*. In general, findings are reported to management within 1 week of the audit and a response is due from management within 30 days.

16.2.2 Performance Audits

Corporate QA may arrange for double blind PT studies to be performed in the laboratories. Results are given to Management and Corrective actions of any findings are coordinated at each facility by the QA Managers and Laboratory Directors/Managers. These studies are performed on an as needed basis. They may be performed when concerns are raised regarding the performance of a particular method in specific laboratories, periodically to evaluate methods that may not normally be covered in the external PT program or may be used in the process of developing best practices. The local QA Manager may also arrange for PT studies on an as needed basis (refer to Section 16.3.2 for additional information on Performance Audits).

16.2.3 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

16.3 EXTERNAL AUDITS

TestAmerica facilities are routinely audited by clients and external regulatory authorities. External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is the TestAmerica policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. The Laboratory Supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. This time frame is generally 30 days.

Be aware that NELAC requires that the audit response report be acceptable to the primary accrediting authority after the second submittal. The lab shall have accreditation revoked for all or any portion of its scope of an accreditation for any or all fields of testing, a method, or analyte within a field of testing if it is not corrected.

TestAmerica North Canton cooperates with clients and their representatives to monitor laboratory performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

16.3.1 Confidential Business Information (CBI) Considerations

During on-site audits, on-site auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

16.3.2 Performance Audits

The laboratory is involved in performance audits conducted as directed by state agencies through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies—water pollution and soil studies.

- It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Further, where PT samples present special or unique problems in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.
- PTs generally do not have holding times associated with them. In the absence of any holding time requirement, it is recommended that the holding time begin when the PT sample is prepared according to the manufacturer's instructions. Holding times should apply to full volume PT samples only if the provider gives a meaningful "sampling date". If this is not provided, it is recommended that the date/time of opening of the full volume sample be considered the beginning of holding time.
- Login will obtain the COC information from the documentation provided with the PTs with review by QA or other designated staff.

- Vials will be prepared as required in the instruction set provided with the samples. After preparation to full volume the sample may be spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.
- PT samples will not undergo multiple preps, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, UNLESS this is what would be done to a normal client sample. An example of this is if a client requests, as PT clients do, that we split VOA coeluters, then dual analysis IS normal practice.
- The type, composition, concentration and frequency of quality control samples analyzed with the PT samples shall be the same as with routine environmental samples.
- Instructions may be included in the laboratory SOPs on how low-level samples are analyzed, including concentration of the sample or adjustment of the normality of titrant. When a PT sample falls below the range of the routine analytical method, the low-level procedure may be used. Results below the routine reporting limit are reviewed against PT instructions and analyte PT reporting limit values.
- No special reviews shall be performed by operation and QA, UNLESS this is what would be done to a normal client sample. To the degree that special report forms or login procedures are required by the PT supplier, it is reasonable that the laboratory WOULD apply special review procedures, as would be done for any client requesting unusual reporting or login processes.
- Written responses to unacceptable PT results are required. In some cases, it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

16.4 AUDIT FINDINGS

Internal or External Audit findings should be documented using the corrective action process. The laboratory is expected to prepare a response to audit findings within 30 days of receipt of an audit report unless the report specifies a different time frame. The response may include action plans that could not be completed within the 30-day timeframe. In these instances, a completion date must be set and agreed to by Operations Management and the QA Manager.

Responsibility for developing and implementing corrective actions to findings is the responsibility of the Group Leader where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory must take timely corrective action, and must notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

The procedures must be in accordance to SOP CA-L-S-001, Internal Investigations of Data Discrepancies and Determination of Data Recall.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

Figure 16-1.

Example - Internal Audit Workbook

				TestAmerica <Name> Last Updated: 9/10/2007		
THE LEADER IN ENVIRONMENTAL TESTING				Workbook Instruction No. CA-Q-WI-011		
Internal Audit Workbook Summary Page						
Note: Click on the (Summary Page) to located on each audit sheet to return to this page.						
* The lab may choose to audit these areas with each method/analyst/instrument audit. The auditor must document on the checklist that this item is audited as part of the <defined> audit.						
	Area Audited	Audit Type	Audit Cycle	Scheduled	Date Audited	Date Closed
	(Click on the Area to get to that Spreadsheet)					
1	Balances	System	6 mo			
2	Temperature Logs/Thermometers	System	6 mo			
3	Sample Storage and Disposal	System	1 yr			
4	Maintenance Logs *	System	6 mo			
5	Volatile Storage Blanks	System	6 mo			
6	Lab Water Quality Testing	System	6 mo			
7	Sample Log In	System	1 yr			
8	Shipping Procedures	System	1 yr			
9	Computer Operations (LIMS)	System	1 yr			Pending Corp. IT Policies
10	SOP & Document Distribution System	System	1 yr			
11	Archiving Electronic & Paper Records	System	1 yr			Pending Corp. IT Policies
12	Statistical Process Control	System	1 yr			
13	Data Review System	System	1 yr			
14	Final Report Generation	System	1 yr			
15	Standards/Reagents *	System	6 mo			
16	Manual Integration *	System	1 yr			
17	Corrective Action System	System	1 yr			
18	Training Records	System	6 mo			
19	MDLs	System	1 yr			
20	SOPs - Prep/Review/Update Process	System	1 yr			
21	Purchasing/Procurement	System	1 yr			
22	Eppendorf/Diluter/Dispenser Calibration Check	System	6 mo			
23	Subcontract Lab Approval	System	1 yr			
24	Customer Complaint System	System	1 yr			
25	Methods	Method	2 yr			
			
			
			

Figure 16-2.

Example – Internal Audit System Checklist: Corrective Actions



(Summary Page)

TestAmerica <Location>

INTERNAL AUDIT - Corrective Actions

[Printed Name(s) or Date(s)]

Area Audited: _____
Auditor: _____
Date: _____
Persons Contacted During Audit: _____
Date Reported to Department Manager: _____
Reported To: _____
Date Reported to Lab Director/Manager: _____
Reported To: _____
Date Response Due: _____
Response Received and Accepted by QA Manager: _____
Associated Corrective Action Report Number(s): _____
Scheduled Follow-up: _____

Item	Requirement	Ref.	Y	N	NA	Evidence/Comments	Follow Up
1	Does the laboratory have a corrective action program in place?	5.4.10.1					
2	Does the laboratory have a current corrective action SOP or is this information in the QA Manual?	5.4.10.1					
3	Do all laboratory personnel have documented training and access to initiate corrective actions?	5.4.10.1					
4	Are causes clearly identified by department, staff name, scope of issue (how many reports affected)?	5.4.10.6					
5	Is a root cause for the issue identified?	5.4.10.2					
6	Is a corrective action (plan) clearly described?						
7	Was the corrective action fully implemented?						
8	Is documentation (if applicable) completed as specified by the corrective action (training, revised SOP, etc)						
9	Has a follow-up assessment been conducted to verify the corrective action was successful?						
10	Are corrective actions reviewed on a regular basis by management?	5.4.10.6a 5					
11	Is there a defined distribution flow for corrective action notification, review, closure, and follow-up?	5.4.10.6a					
12	Are non-conformances reviewed on a regular basis and used, if necessary, to initiate root cause corrective actions?						
13	Does the lab have a documented procedure for QC corrective action (i.e., documented within each method / parameter SOP or in the QA Manual)?	4.10.1					
14	Verify Corrective Actions from previous systems audits. List Items:						
15							
16							
17							

Auditor Signature: _____

Primary Reference(s): Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices
NELAC Standard, June 2003
DoD Quality Systems Manual, Version 3, January 2006
EPA Manual for the Certification of Laboratories Analyzing Drinking Water

SECTION 17

MANAGEMENT REVIEWS (NELAC 5.4.14)

17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director/Manager for review and comments. The final report shall be submitted to the Laboratory Director as well as the appropriate Quality Director and General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. At a minimum, the report content will contain the items listed below. During the course of the year, the Laboratory Director/Manager, General Manager or Corporate QA may request that additional information be added to the report.

The TestAmerica QA Report template is comprised of a discussion of three key QA issues facing the laboratory and ten specific sections (Figure 17-1):

- **Metrics:** Describe actions or improvement activities underway to address any outlying quality metrics that have been reported in the monthly Quality System Metrics Table.
- **Quality System Metrics Table:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).
- **SOPs:** Report SOPs that have been finalized and report status of any outstanding SOP reviews.
- **Corrective Actions:** Describe highlights and the most frequent cause for report revisions and corrective/preventive action measures underway. Include a discussion of any recalls handled at the lab level as per Section 6.2.2 in the Investigation/Recall SOP, CA-L-S-001. Include a section for client feedback and complaints. Include both positive and negative feedback. Describe the most serious client complaints and resolutions in progress.
- **MDLs and Control Limits:** Report which MDLs/ MDL verifications are due. Report the same for Control Limits.
- **Audits:** Report Internal and External Audits that were conducted. Include all relevant information such as which methods, by whom, corrective actions needed by when and discuss unresolved audit findings.
- **Performance Testing (PT) Samples:** Report the PT tests that are currently being tested with their due dates, report recent PT results by study, acceptable, total reported and the month and year.
- **Certifications:** Report on any certification programs being worked on by due date, packages completed. Describe any issues, lapses, or potential revocations.
- **Regulatory Updates:** Include information on new state or federal regulations that may impact the laboratory. Report new methods that require new instrumentation, deletion of methods, changes in sampling requirements and frequencies etc...

- **Miscellaneous:** Include any issues that may impact quality within the laboratory.
- **Next Month:** Report on plans for the upcoming month.
- **Lab Director Comments Section:** This section gives the Laboratory Director/Manager the opportunity to comment on issues discussed in the report and to document plans to resolve these issues. Unresolved issues that reappear in subsequent monthly reports must be commented on by the Laboratory Director/Manager.
- **Quality System Metrics Table:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The VP-QA/EHS prepares a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Analytical Division Senior Management Team and General Managers.

17.2 ANNUAL MANAGEMENT REVIEW

The Senior Lab Management Team (Laboratory Director, Technical Director, Operations Manager, QA Manager, HR Supervisor, I.T. Supervisor) conducts an annual review of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director/Manager. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This review uses information generated during the preceding year to assess the “big picture” by ensuring that routine quality actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review (refer to Section 17.1) should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review
- Prior Monthly QA Reports issues
- Laboratory QA Metrics
- Review of report reissue requests
- Review of client feedback and complaints
- Issues arising from any prior management or staff meetings
- Minutes from prior Senior Management team meetings. Issues that may be raised from these meetings include:

- Adequacy of staff, equipment and facility resources
 - Adequacy of policies and procedures
 - Future plans for resources and testing capability and capacity
- The annual internal double blind PT program sample performance (if performed)
 - Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

The annual review includes the previous 12 months. Based on the annual review, a report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants
- A reference to the existing data quality related documents and topics that were reviewed
- Quality system or operational changes or improvements that will be made as a result of the review, e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)

The QA Manual is also reviewed at this time, and revised to reflect any significant changes made to the quality systems.

17.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The Corporate Data Investigation/ Recall SOP shall be followed (SOP CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Chairman/CEO, President/CEO, COOs, and Quality Directors receive a monthly report from the VP of Quality and EHS summarizing any current data integrity or data recall investigations as described in SOP CA-L-S-001. The General Managers are also made aware of progress on these issues for their specific labs.

Figure 17-1.

Example - QA Monthly Report to Management

LABORATORY: x
PERIOD COVERED: Month/Year
PREPARED BY: x DATE: Month Day, Year
DISTRIBUTED TO: xx (Include LD, GM, QA Director, etc...)

THREE KEY ISSUES FOR MONTH:

Include a discussion of three key issues that were focused in on this month.

1. x
 2. x
 3. x
-

1. METRICS

Describe actions or improvement activities underway to address any outlying quality metrics.

2. SOPs

See Tab for SOP specifics.

The following SOPs were finalized (or reviewed for accuracy): (See Tab)

The following SOPs are due to QA: xx

In QA to complete: xx

3. CORRECTIVE ACTION

Highlights: xx

Revised Reports:

Describe the most frequent cause for report revisions and corrective/preventive action measures underway.

Data Investigations/Recalls (Corporate Data Investigation/Recall SOP) :

Include a discussion of any recalls handled at the lab level as Corp SOP.

Client Feedback and Complaints:

Include both positive and negative feedback.

Describe the most serious client complaints) and resolutions in progress.

4. MDLs AND CONTROL LIMITS

MDLs Due:

Control Limits Due:

5. AUDITS

INTERNAL AUDITS

Discuss Any Outstanding Issues (or Attach Summary):

EXTERNAL AUDITS

Discuss Any Outstanding Issues (or Attach Summary):

6. PT SAMPLES

The following PT samples are now in house (Due Dates):

xx

7. CERTIFICATIONS

Certification Packages Being Worked On (Include Due Date):

x

Describe any issues, lapses, or potential revocations.

8. REGULATORY UPDATE

Include information on new state or federal regulations that may impact the laboratory – new methods that require new instrumentation, deletion of methods, changes in sampling requirements or frequencies, ...

9. MISCELLANEOUS

Include any issues that may impact quality within the laboratory.

10. NEXT MONTH

Items planned for next month.

LAB DIRECTOR COMMENTS AND PLANNED CORRECTIVE ACTIONS:

LAB DIRECTOR REVIEW:

DATE:

Figure 17-2.

Example - Laboratory Metrics Categories

No. of reports for month
No. of reports revised due to lab error
Percent revised reports
No. of Data Recall Investigations
No. of reports actually recalled
No. of Corrective Action Reports
No. of Corrective Action Reports still open
Total number of unresolved open Corrective Action Reports
Percent of unresolved open Corrective Action Reports
No. of reports independent QA reviewed
Percent QA Data Review: Reports
No. of technical staff (analysts/technicians, including temps)
No. of analyst work product reviewed year-to-date
No. of analytical instruments with electronic data file storage capability
No. of Analytical instruments reviewed for data authenticity year-to-date
Percent Analyst/Instrument Data Authenticity Audits
No. of client complaints
No. of client compliments
No. of planned internal audits
No. of planned internal method audits performed year-to-date
Percent annual Internal Audits complete
No. of open Internal Audit findings past due
Total Number of External Audit findings
No. of open External Audit findings past due
Percent External Audit findings past due
No. of PT analytes participated and received scores
No. of PT analytes not acceptable
Percent PT cumulative score
No. of PT repeat analyte failures cumulative (analyte failed more than once in 4 consecutive studies by PT type--only applies to failed analytes)
No. of SOPs

No. of SOPs reviewed/revised within 24 months
No. of methods or administrative procedures without approved SOPs
SOP status
Method certification losses due to performance/audit issues
Hold Time violations due to lab error
Date of last Comprehensive Ethics Training session
No. of staff that haven't received Comprehensive Ethics Training (>90 days from employment date)
MDL status (good, fair, or poor) >90%, >70%, <70%
Training Documentation Records (good, fair, or poor)
LQM Revision/review date
QAM updated to new integrated template
Last annual Internal Audit date (opened, closed)
Last Management QS Review date
No. of SOPs required for 12-month review cycle (DOD or drinking water)
No. of SOPs for 12-month cycle/revised within 12 months (<i>Includes QS and Methods Listed in QSM</i>)
12-month percent SOP status (<i>Includes QS and Methods Listed in QSM</i>)

SECTION 18

PERSONNEL (NELAC 5.5.2)

18.1 OVERVIEW

TestAmerica's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Appendix 2.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

18.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

TestAmerica makes every effort to hire analytical staff that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. There are competent analysts and technicians in the industry who have not earned a college degree. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are

located on the TestAmerica intranet "Human Resources" web-page (also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc. are also considered

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – General	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Director – Wet Chem only (no advanced instrumentation)	Associate degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

Specialty	Education	Experience
Technical Director - Microbiology	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

18.3 **TRAINING**

TestAmerica is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame*	Employee Type
New Hire Orientation	Immediately	All
Environmental Health & Safety Orientation	Day of hire	All
Environmental Health & Safety Orientation Follow-up Test	30-60 days after hire	All
Environmental Health & Safety Training	Refer to EH&S Manual	All
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 20.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in the employee's training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct, e.g., ethics. This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in the Laboratory Training SOP CORP-QA-0013, Employee Orientation and Training.

18.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times, TestAmerica has established an Ethics Policy CA-L-P-001 and an Ethics Statement/Agreement (Appendix 1). All initial and annual training is documented by signature on the signed Ethics Statement/Agreement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts; and for that reason, TestAmerica has a zero tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting
- Ethics Policy (Appendix 1)
- How and when to report ethical/data integrity issues. Confidential reporting.

- Record keeping
- Discussion regarding data integrity procedures
- Specific examples of breaches of ethical behavior--peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion
- Internal monitoring. Investigations and data recalls
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient

Additionally, a Data Integrity Hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 19

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)

19.1 OVERVIEW

TestAmerica North Canton is an 11,466 sq ft secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity-controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

19.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. A 225KVA UPS is installed in the main electrical bus to provide at least 15 minutes of backup power in the event of a power failure. This unit also provides voltage and frequency control of lab and office power. A spike/surge arrestor is installed to protect against power surge/sag and lightning strikes. A 30 KW natural gas-fueled backup generator is installed to provide power to the I.T. area in the event of a power failure. Additionally, this generator provides power to two walk-in sample

storage coolers and several other smaller sample storage coolers. Smaller portable generators are available to provide “spot power” where needed in the event of a power failure.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels (refer to Section 12).

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

19.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

19.4 FLOOR PLAN

A floor plan can be found in Appendix 3.

19.5 BUILDING SECURITY

Building keys are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of TestAmerica North Canton. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

SECTION 20.0

TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)

20.1 OVERVIEW

TestAmerica North Canton uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples; and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

20.2 STANDARD OPERATING PROCEDURES (SOPs)

TestAmerica North Canton maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for preparation, review, revision, and control are incorporated by reference to SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP), and SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures.
- SOPs are reviewed at a minimum of every two years (annually for Drinking Water and DoD SOPs); and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP. Refer to the corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure, and SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures, for content and requirements of technical and non-technical SOPs.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from

the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

20.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services, e.g., special matrices, non-routine compound lists, etc., the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

20.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

In general, TestAmerica North Canton follows procedures from the referenced methods shown below in Section 20.3.1.4.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

20.4.1.1 The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999
- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995. Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.

- *Standard Methods for the Examination of Water and Wastewater*, 18th/19th/20th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)*, Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.
- *Annual Book of ASTM Standards*, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

TABLE 20-1. Wet Chemistry Methods ¹

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Acidity	Water		305. ² SM 2310 B	--	--
Alkalinity, Bicarbonate, Carbonate	Water		305. ² SM 2320 B		
	Solid		EPA 310.1 ² (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water		--	EPA 7063	--
Ash Content	Solid		--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water		EPA 405.1 SM 5210 B	--	--
Bromide	Water		EPA 300.0A	EPA 9056A	--
	Waste		EPA 300.0A	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A	--
Cation-Exchange Capacity	Solid		--	EPA 9081	--
Chemical Oxygen Demand	Water		EPA 410.4 SM 5220D	--	--
	Waste		EPA 410.4	--	--
Chloride	Water		EPA 300.0A EPA 325.2 ²	EPA 9056A EPA 9251	EPA 325.2 ²
	Waste		EPA 300.0A	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A EPA 9251(M)	--

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Chromium, Hexavalent	Water		EPA 3500-Cr-D	EPA 7196A	--
	Waste		EPA 3500-Cr-D	EPA 7196A	--
	Solid		--	EPA 3060A EPA 7196A	--
Specific Conductance	Water		EPA 120.1 SM 2510B	EPA 9050A	--
	Waste		EPA 120.1	EPA 9050A	--
	Solid		--	EPA 9050A	--
Chlorine, Residual	Water		EPA 330.5 ² SM 3500 CL-G	--	--
Cyanide (Amenable)	Water		EPA 335.1 ² SM 4500 CN-G	EPA 9012A	--
	Solid		--	EPA 9012A	--
Cyanide (Total)	Water		SM 4500-CN E EPA 335.4	EPA 9012A	---
	Waste		--	EPA 9012A	--
	Solid		--	EPA 9012A	--
Cyanide (Weak and Dissociable) (Free)	Water		SM 4500-CN I	--	--
Dissolved Oxygen	Water		360.1 ² SM 4500 O-G	--	--
Flash Point	Waste		--	EPA 1010	ASTM D93-9
	Solid		--	EPA 1010	ASTM D93-9
Fluoride	Water		EPA 300.0A EPA 340.2 ²	EPA 9056A	SM 4500 F-C, ISE
	Waste		EPA 340.2 (M) ² EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 340.2 (M) ² EPA 300.0A (M)	EPA 9056A	--
Iron, Ferrous & Ferric	Water		SM 3500 FE D	--	--
Hardness	Water		EPA 130.2 ²	--	SM 2340B
Moisture	Solid		---	EPA 160.3 (M) ASTM D2216-90	---
Nitrogen, Ammonia	Water		EPA 350.1	--	EPA 350.2 ²
	Waste		EPA 350.1	--	EPA 350.2 ²
	Solid		EPA 350.1	--	EPA 350.2 ²
	Water		SM 4500 NH ₃ -E (Titration)	--	--

Analytical Parameters Matrix	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Nitrogen, Ammonia (cont'd)	Water		SM 4500 NH ₃ -F (ISE)	--	--
Nitrite (NO ₂)	Water		EPA 300.0A	EPA 9056A	--
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A	--
Nitrate (NO ₃)	Water		EPA 300.0A	EPA 9056A	SM 4500 NO ₃ -E
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M)	--	--
Nitrate plus Nitrite NO ₂ /NO ₃	Water		EPA 353.2	--	--
	Waste		EPA 353.2	--	--
Total Kjeldahl Nitrogen (TKN)	Water		EPA 351.3	--	SM 4500 NO ₃
	Waste		EPA 351.3	--	--
	Solid		EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water		EPA 1664A	EPA 9071B	--
	Waste		EPA 1664A	EPA 9071B	--
	Solid		--	EPA 9071B	--
Ortho-phosphate o-PO ₄	Water		EPA 300.0A EPA 365.1	EPA 9056A	SM 4500 P-E
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M) EPA 365.1	EPA 9056A	--
pH	Water		EPA 150.1 ²	EPA 9040B	EPA 9041
	Waste		SM 4500 H-B	EPA 9045C	--
	Solid		---	EPA 9045C	--
Paint Filter	Water		--	EPA 9095A	--
Phenolics	Water		EPA 420.1	--	--
	Waste		--	EPA 9065	--
	Solid		--	EPA 9065	--
Phosphorus (Total)	Water		EPA 365.1	--	SM 4500 P-E
	Waste		EPA 365.1	--	--
	Solid		EPA 365.1	--	--
Sulfate (SO ₄)	Water		EPA 300.0A EPA 375.4 ²	EPA 9056A EPA 9038	--
	Waste		EPA 300.0A (M) EPA 375.4 ²	EPA 9056A EPA 9038	--
	Solid		EPA 300.0A (M)	EPA 9056A EPA 9038 (M)	--

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA	Other
Sulfide	Water		EPA 376.1 ²	EPA 9030A SM 4500	9030B/9034
Total Organic Carbon (TOC)	Water		EPA 415.1 ²	EPA 9060	SM 5310 D
	Waste		--	EPA 9060	--
	Solid		EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water		--	EPA 9020B EPA 9023(EOX)	EPA 450.1
	Waste		--	--	--
	Solid		--	EPA 9020B	--
Total Petroleum Hydrocarbons	Water		EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste		EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid		--	EPA 9071B	--
Total Solids	Water		EPA 160.3	--	--
	Waste		EPA 160.3	--	--
	Solid		EPA 160.3 (M)	--	--
Total Dissolved Solids	Water		EPA 160.1	--	2540E
Total Suspended Solids	Water		EPA 160.2	---	2540E
Volatile and Volatile Suspended Solids	Water		EPA 160.4	--	--
Settleable Solids	Water		EPA 160.5	--	--
Turbidity	Water		EPA 180.1	--	--

¹ Any matrix not listed is not applicable for the associated method

² Removed from 40CFR

TABLE 20-2. Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water		EPA 245.1	EPA 7470A	--
	TCLP Leachate		--	EPA 7470A	--
	Waste		--	EPA 7471A	--
	Solid		EPA 254.5	EPA 7471A	--

TABLE 20-3. Methods for Mercury by Cold Vapor Atomic Fluorescence

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water		EPA 245.7	--	EPA 1631E

TABLE 20-4. Methods for Metals by ICP and ICPMS

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Aluminum	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Antimony	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Arsenic	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Barium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Beryllium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Boron	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---
Calcium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---
Cadmium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Cobalt	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Chromium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Copper	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Iron	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Lead	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Magnesium	Water		EPA 200.7	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7	EPA 6010B EPA 6020	---
Manganese	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Molybdenum	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Nickel	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Potassium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Selenium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Silver	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Sodium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Tin	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Thallium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Titanium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Vanadium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		---	EPA 6010B EPA 6020	---
Zinc	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

TABLE 20-5. Metals Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water		---	EPA 1311	---
	Waste		---	EPA 1311	---
	Solid		---	EPA 1311	---
ICP Metals	Water		EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
ICPMS Metals	Water		EPA 200.8	EPA 3010A	---
	TCLP		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
CVAA Mercury	Water		EPA 245.1	EPA 7470A	---
	TCLP Leachate		---	EPA 7470A	---
	Waste		---	EPA 7471A	---
	Solid		---	EPA 7471A	---
CVAFS Mercury Low Level	Water		EPA 245.7	---	EPA 1631E

TABLE 20-6. Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water		EPA 624	EPA 5030B	---
	Waste		---	EPA 5030B EPA 5035	---
	Solid		---	EPA 5035 EPA 5035A	---
Halogenated Volatiles by GC	Water		EPA 601	EPA 5030B	---
	Waste		---	EPA 5030B EPA 5035	---
	Solid		---	EPA 5035 EPA 5035A	---
Aromatic Volatiles by GC	Water		EPA 602	EPA 5030B	---
	Waste		---	EPA 5030B EPA 5035	---
	Solid		---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water		EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate		---	EPA 3510C EPA 3520C	---
	Waste		---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid		---	EPA 3550B EPA 3540C EPA 3541	---
Pesticides/PCBs by GC	Water		EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate		---	EPA 3510C EPA 3520C	---
	Waste		---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid		---	EPA 3550B EPA 3540C EPA 3541	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Herbicides by GC	Water		EPA 615	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water		---	EPA 5030B	WI GRO
	Waste		---	EPA 5030B EPA 5035	WI GRO
	Solid		---	EPA 5035 EPA 5035	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water		---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate		---	EPA 3510C EPA 3520C	---
	Waste		---	EPA 3550B EPA 3580A	WI DRO
	Solid		---	EPA 3550B	WI DRO

TABLE 20-7. Organic Methods of Analysis

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water		EPA 624	EPA 8260B	---
	Waste		---	EPA 8260B	---
	Solid		---	EPA 8260B	---
Halogenated Volatiles by GC	Water		EPA 601	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Aromatic Volatiles by GC	Water		EPA 602	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Semivolatiles by GC/MS	Water		EPA 625	EPA 8270C	
	Waste		---	EPA 8270C	---
	Solid		---	EPA 8270C	---
Pesticides/PCBs by GC	Water		EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate		---	Pesticides 8081A PCBs 8082	---
	Waste		---	Pesticides 8081A PCBs 8082	---
	Solid		---	Pesticides 8081A PCBs 8082	---
Phenoxyacid Herbicides by GC	Water		---	EPA 8151A	---
	TCLP Leachate		---	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Gasoline Range Organics by GC	Water		---	EPA 8015B (M)	WI GRO
	Waste		---	EPA 8015B (M)	---
	Solid		---	EPA 8015B (M)	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID	Water		---	EPA 8015B (M)	WI DRO
	Waste		---	EPA 8015B (M)	---
Dissolved Gases RSK-175	Water		---	---	SOP
Formaldehyde Carbonyl Compounds	Water		---	EPA 8315	---

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

20.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

- 20.4.2.1 A demonstration of capability is performed whenever there is a change in instrument type, method or personnel.
- 20.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures. Refer to Section 15, Control of Records.
- 20.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method).
- The reporting limit is set at or above the first standard of the curve for the analyte.
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. This must be addressed in the Case Narrative.

- Refer to Section 12, Control of Non-Conforming Work.

20.4.3 Initial Demonstration of Capability (IDOC) Procedures

- 20.4.3.1 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).
- 20.4.3.2 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest. Refer to SOP CORP-QA-0013, Employee Orientation and Training, for details on this procedure.

A certification statement (see Figure 20-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP/Methods Manual (Section 20.2) and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method. The information included in the checklist below (Figure 20-2) is needed before samples are accepted for analysis by a new method.

20.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled (from 2003 NELAC Standard).

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

20.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

20.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some

cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

20.6.1.2 **Determination of Method Sensitivity**

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determinations of MDLs are described in Section 20.6.

20.6.1.3 **Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)**

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

20.6.1.4 **Determination of Interferences**

A determination that the method is free from interferences in a blank matrix is performed.

20.6.1.5 **Determination of Range**

Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

20.6.1.6 **Determination of Accuracy and Precision**

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

20.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

20.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

20.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B, or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to Section 20.7.10). The analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

20.7.1 MDLs are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory will use the highest calculated MDL for all instruments used for a given method as the MDL for reporting purposes. This MDL is not required for methods that are not readily spiked, e.g., pH, turbidity, etc. Titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.

20.7.2 MDLs must be run against acceptable instrument QC, including ICVs and Tunes. This is to ensure that the instrument is in proper working condition and falsely high or low MDLs are not calculated.

20.7.3 Use only clean matrix which is free of target analytes (e.g.: Laboratory reagent water, Ottawa Sand) unless a project specific MDL is required in a field sample matrix.

20.7.4 The Reporting Limit should generally be between two and five times the MDL. If the MDL is being performed during method development, use this guideline to determine the Reporting Limit for the analysis. For DoD labs, the RL is $\geq 3x$ MDL. If a sample is diluted, the reported MDL is adjusted according to the dilution factor.

20.7.5 If the MDL is $< 1/10$ of the spike concentration for more than 10% of the analytes in the method ($< 1/5$ of spike recovered for DoD for water samples), the MDL must be repeated (including extraction or digestion) using a lower spike level unless the percent recovery is $< 50\%$ or $> 150\%$ of the “true value”. Note: The concentration of the spike will be at a level below the calibration range. Note: The spiking concentration must be less than the reporting limit.

20.7.6 The calculated MDL cannot be not greater than the spike amount.

20.7.7 If the most recent calculated MDL does not permit qualitative identification of the analyte then the laboratory may use technical judgment for establishing the MDL (e.g., calculate what level would give a qualitative ID, compare with IDL (20.7), spike at a level where qualitative ID is determined and assign that value as MDL, minimum sensitivity requirements, Standard deviation of method blanks over time, etc.).

20.7.8 Each of the seven spikes must be qualitatively identifiable, e.g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc. Manual integrations to force the baseline for detection are not allowed.

20.7.9 The initial MDL is calculated as follows:

$$\text{MDL} = t_{(n-1, 1-a = 0.99)} \times (\text{Standard Deviation of replicates})$$

where $t_{(n-1, 1-a = 0.99)} = 3.143$ for seven replicates.

20.7.10 Subsequent to the initial MDL determination, periodic MDL verification, confirmation or determinations may be performed by the procedure in 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices (e.g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.). The procedures utilized must be documented in the MDL SOP NC-QA-0021, Evaluation of Method Detection Limits for Chemical Tests.

20.7.11 Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL. Any result that falls between the MDL and the Reporting limit, when reported, will be qualified as an estimated value.

20.7.12 Detections reported down to the MDL must be qualitatively identified.

20.7.13 MDLs and Reporting limits are adjusted in LIMs based on moisture content and sample aliquot size.

20.8 INSTRUMENT DETECTION LIMITS (IDL)

20.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

20.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either by using seven replicate spike analyses, like MDL but without sample preparation, or by the analysis of ten instrument blanks and calculating three times the absolute value of the standard deviation.

20.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

20.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

20.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately two times the calculated MDL. The analytes must be qualitatively identified or see Section 20.6.7 for other options. This verification does not apply to methods that are not readily spiked, e.g., pH, turbidity, etc. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established (see Section 20.6.7). MDLs must be verified at least quarterly.

20.9.2 When a Reporting limit is established, it must be initially verified by the analysis of a low-level standard or QC sample (LCS at 1-2 the reporting limit) and annually thereafter. Unless there are requirements to the contrary, the acceptance criteria is $\pm 50\%$. The annual requirement is waived for methods that have an annually verified MDL.

20.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept in each department.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

Note: Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes, e.g., m-xylene and p-xylene, and are quantitated and reported as a single analyte, e.g., m,p-xylenes.

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, he or she uses a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24-hr period for 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time ± 3

Standard Deviations. A peak outside the retention time window will not be identified by the computer as a positive match of the analyte of interest.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be used, e.g., for 8000 series methods a default of 0.03 minutes may be used, and EPA CLP 0.05 minutes is used. The same concept is applied when any peak outside of that window will not be identified by the computer as a positive match.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window, but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

20.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, atomic absorption, or fluorescence profiles.

20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

20.12.1 Uncertainty is “a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand” (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result’s validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an “expanded uncertainty”: the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor $k=2$.

20.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

20.12.3 The uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

20.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.

20.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement, e.g., 524.2, 525, etc., and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

20.13 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

20.13.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the QuantIMS LIMS which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes AS400 which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

20.13.1.1 Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

Note: "Commercial off-the-shelf software in use within the designed application range is considered to be sufficiently validated" from *NELAC 2003 Standard*. However, laboratory specific configurations or modifications are validated prior to use.

- In order to assure accuracy, all data entered or transferred into the LIMS data system goes through a minimum of two levels of review.
- The QA department performs random data audits to ensure the correct information has been reported.

- Changes to reports are documented via Change Order Forms and by noting "Revised" on the cover page.
- Analytical data file security is provided through three policies.
 - The first policy forbids unauthorized personnel from using laboratory data acquisition computers.
 - The second policy is the implementation of network passwords and login names that restrict directory access.
 - The third layer is maintained through the LIMS and includes the use of username/password combinations to gain access to the LIMS system, the fact that all data in the LIMS is associated with the user to added/reviewed the data, and the restriction of review authority of data.
- All software installations will be in accordance with any relevant copyright licensing regulations.
- All software installed on any computer within the laboratory must be approved by the Information Technology Department regional support technician assigned to the laboratory. Shrink-wrapped or otherwise sealed OEM software that is directly related to instrument usage does not need approval, but the Information Technology Department must be notified of the installation.
- Anti-virus software shall be installed on all servers and workstations. The anti-virus software shall be configured to check for virus signature file and program updates on a daily basis and these updates will be pushed to all servers and workstations. The anti-virus software will be configured to clean any virus-infected file if possible, otherwise the file will be deleted. Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.
- **Interlab LIMS Permissions Policy**
 - PURPOSE - The purpose of this policy is to provide a mechanism for maintaining the integrity of information contained in each laboratory's LIMS while providing the necessary access for information sharing to staff at other laboratory facilities.
 - DEFINITIONS - Host Laboratory: The laboratory facility that 'owns' the LIMS system or 'hosts' a project/job.
 - POLICIES
 - (a) All permissions for the laboratory's LIMS system must only be granted by a representative of that laboratory.
 - If someone outside of the host lab needs permissions for Project Management or other uses, they must go through the Lab Director or his/her designated representative.
 - Permissions must never be granted without the knowledge of the host laboratory.
 - (b) Only laboratory analytical or QA staff from the home laboratory may have edit permissions for laboratory analysis data.

(c) Any changes made in laboratory's LIMS system:

- Must be documented and traceable.
- If made by staff of an affiliate lab, written permission from the home lab to make the changes (email approval is sufficient) is required.
- No corrections may be made in another laboratories system without their knowledge.

(d) Data qualifiers in laboratory reports must only be corrected, edited, etc. by the staff at the host laboratory.

(e) Full analytical data "View" only permissions may be granted to outside Project Management and Sales staff. Search permissions may also be granted so status may be checked.

(f) All qualifiers must be approved by QA staff before adding to standard reference tables.

(g) Please contact Corporate QA or IT staff if you have any questions regarding implementation or interpretation of this policy.

20.13.1.2 **Ensure Information Availability:** Protection against loss of information or service through scheduled back-ups, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

- Insured by timely backup procedures on reliable backup media, stable file server network architecture, and UPS protection
- UPS Protection:
 - Each fileserver is protected by an appropriate power protection/backup unit. In the event of a power outage, there is approximately 15-30 minutes of up-time for the servers prior to shutdown. This allows for proper shutdown procedures to be followed with the file servers.
- File Server Architecture
 - All files are maintained on multiple Windows 2000 or newer servers which are secured physically in the Information Technology office. Access to these servers is limited to members of the Information Technology staff.
 - All supporting software is maintained for at least five years from the last raw data generated using that software. Length of time is dependent on local regulations or client requirements, e.g., OVAP requires ten years.
- System Back-up Overview and Procedures
 - Data from both servers and instrument attached PC's are backed up and purged in compliance with the corporate back-up policy.
 - A Maintenance Plan has been defined to create a daily archive of all data within the LIMS database to a backup location. This backup is initiated automatically by either the database or back-up system.

- Backup tapes will be stored in compliance with the corporate Data Backup Policy. Backup verifications are carried out in accordance with the corporate Data Backup Policy.
- Instrument data back-ups are verified on a periodic basis by the QA department when performing electronic data audits. The audit takes place on data that has been moved to a back-up location ensuring that it has been moved.

20.13.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

- All servers are located in a secure area of the IT department offices. Access to the servers is limited to IT staff members.
- The company website contains SSL (Secure Socket Layer) encryption for secure website sessions and data transfers.
- Electronic documents such as PDF files and electronic data deliverables may be made available to clients via the secure web site. The logon page for this web site contains an agreement that the customer must accept before they will be logged on which states that the customer agrees not to alter any electronic data made available to them.

20.13.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved, e.g., extractions, dilutions, instrument readings and concentrations. The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by peer review once updated in LIMS. The review checklists are signed by both the analyst and reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it must not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

20.13.2.1 All raw data must be retained. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

20.13.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter ($\mu\text{g/l}$) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram ($\mu\text{g/kg}$) for solids. The units “mg/l” and “mg/kg” are the same as “parts per million (ppm)”. The units “ $\mu\text{g/l}$ ” and “ $\mu\text{g/kg}$ ” are the same as “parts per billion (ppb).” For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%.

- Several environmental methods, such as color, turbidity, conductivity, use very specific, non-concentration units to report results (e.g., NTU, umhos/cm etc).
- Occasionally, the client requests that results be reported in units which take into account the measured flow of water or air during the collection of the sample. When they provide this information, the calculations can be performed and reported.

20.13.2.3 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

20.13.2.4 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file.

20.13.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out ‘real time’ and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 13.
- Logbooks are controlled by the QA Department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be “Z”d out, signed and dated.
- Worksheets are created with the approval of the QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

20.13.4 Review / Verification Procedures

20.13.4.1 Data Recording Procedures: To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with Section 3 of this document and Policy T-Q-005, Recording Laboratory Observations and Raw Data.

20.13.4.2 Data Reduction and Verification Procedures: Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100% review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed.

20.13.4.2.1 Data Reduction and Initial Verification: Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample amounts, etc.
- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- The appropriate SOPs have been followed and are identified in the project and/or laboratory records
- Proper documentation procedures have been followed
- All non-conformances have been documented
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing

- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to correct misintegrations by an automatic data system software program, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy S-Q-004, Acceptable Manual Integration Practices.

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

If data are directly acquired from instrumentation or a test procedure and processed, or immediately entered into LIMS, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units
- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst.

20.13.4.2.2. Data Verification: Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, supervisor, or designee. This Level 2 check is performed to ensure that Level 1 review has been completed correctly and thoroughly. The second level reviewer examines the data signed by the analyst. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers

- Numerical correctness of calculations and formulas (checked randomly)
- Acceptability of QC data (100% review)
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

This review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and client-specific instructions.
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist.

20.13.4.2.3. Completeness Verification: A third-level review is performed by the reporting and project management staff. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)
- Every parameter or target compound requested is reported with either a value or reporting limit
- All nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory
- The final report contains all the supporting documentation required by the project, and is in either the standard TestAmerica format or in the client-required format.
- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)
- A narrative to accompany the final report will be finalized by the PM. This narrative will include relevant comments collected during the earlier reviews.

20.13.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an

invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines.

- 20.13.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- 20.13.5.2 Analysts shall not increase or decrease peak areas to for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.
- 20.13.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- 20.13.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate-approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 20-1.
Example - Demonstration of Capability Documentation

Analyst Demonstration of Capability **Certification Statement**

Analyst Name
Date:

Test Method:
SOP:
Matrix:

TestAmerica North Canton laboratory
4101 Shuffel Drive NW
North Canton, OH 44720
(330) 497-9396

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the TestAmerica Quality Assurance Plan, has met the Initial or Ongoing Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification following the TestAmerica SOP.
3. A copy of the laboratory-specific SOP is available for all personnel on-site.
4. The data associated with the initial/ongoing demonstration of capability are true, accurate, complete and self-explanatory (*). These data are attached to this certification statement.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/Observations:

Analyst's Name	Signature	Date
Technical Director's Name	Signature	Date
QA Manager's Name	Signature	Date

* *True: Consistent with supporting data.*

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional explanation.

Figure 20-2.

Example - New Method / Additional Analyte Checklist

New Method / Additional Analyte Checklist

The following items are **required** to be completed prior to the acceptance of client samples.

Fill in any blanks that do not apply with "NA".

Forward to Analytical Group, Operations Manager, Technical Director, and QA.

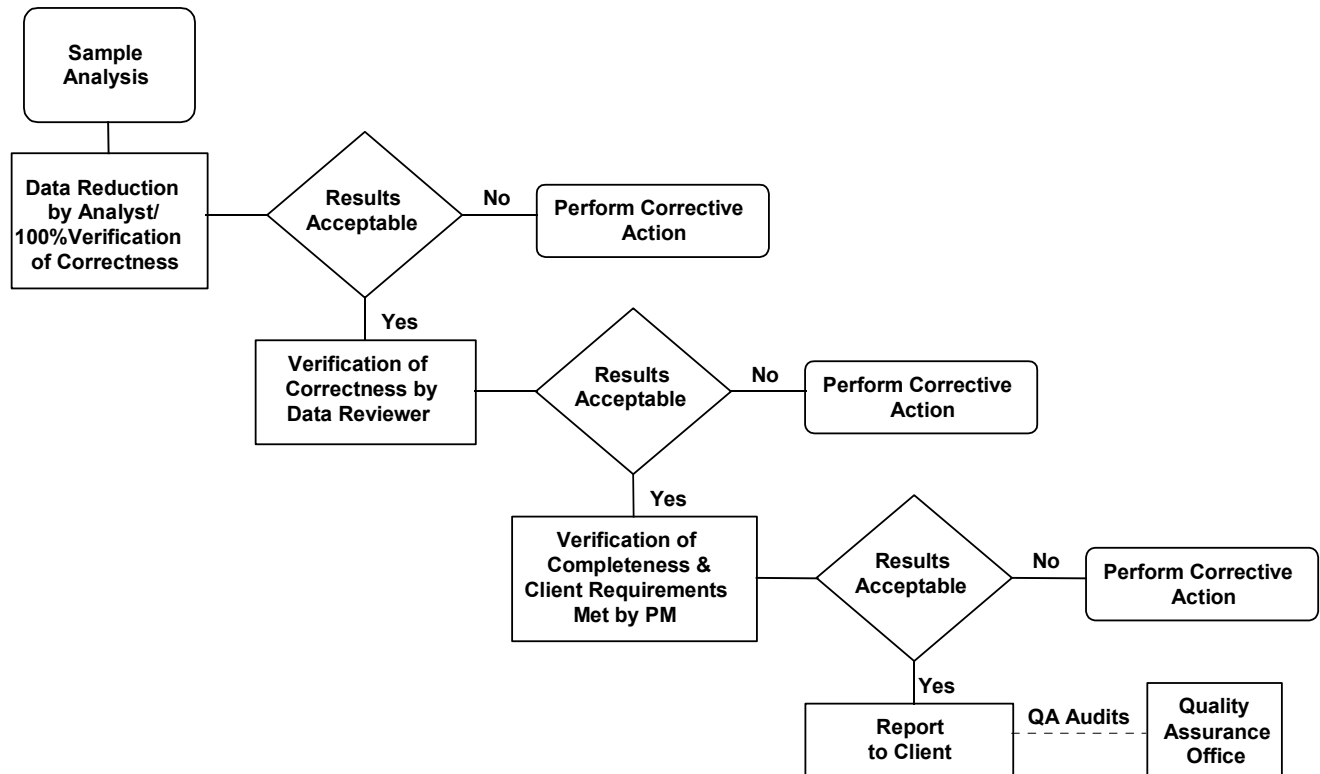
New Method – include method number and reference _____

New Analytes – list analyte and reporting limit

Matrix: water – solid – waste	
Analytical Groups Contacted	
Standard available for new analyte(s), including second source. If not, include time frame for order.	
Cost	
MDL study required	
MDL study completed	
LCS/MS/MSD spike required	
Project tied to QAPP? Lab has a copy?	
Project Program: DoD QSM, LCG, Client specific, State Specific	
Special QC Requirements: control limits, special criteria	
Certification required	
Standard Operating Procedure available for method	
One time project or on-going	
Special reporting parameters	
SAC created in LIMS	
Spike lists created in LIMS	
RL/MDL data entered into LIMS	

QA Review / Acceptance _____ **Date** _____

Figure 20-3.
Work Flow



SECTION 21

EQUIPMENT (AND CALIBRATIONS) **(NELAC 5.5.5)**

21.1 OVERVIEW

TestAmerica purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 21-1.

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

21.2 PREVENTIVE MAINTENANCE

21.2.1 TestAmerica North Canton follows a well-defined program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

21.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

21.2.2.1 Calibrations, routine maintenance, and adjustments are part of the Analyst and Group Leader responsibilities. However, service contracts may be in place for some instruments to cover any major repairs.

21.2.2.2 High purity gases, reagents, and spare parts are kept on hand to minimize repair time and optimize instrument performance.

21.2.3 Table 21-2 summarizes the schedule for routine maintenance. It is the responsibility of each Group Leader to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: For some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

21.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument Maintenance Logbooks may also be used to specify instrument parameters.

21.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

21.2.4.2 Each entry in the instrument log includes the Analyst's initials, date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control, e.g., CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.)

21.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled-in page must be signed across the page entered and the logbook, so it is clear that a page is missing if only half a signature is found in the logbook.

21.2.5 In addition, the maintenance records contain:

- The identification of the instrument/equipment (instrument Serial and Model Number)
- The date the instrument/equipment was put into use.
- If available, the condition when the instrument was received, e.g. new, used, reconditioned

21.2.6 If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses (refer to Sections 12 and 13).

21.2.7 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted using the procedures outlined in Section 8.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

21.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, dispensing devices, if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

21.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to use with at least two certified ASTM Type 1 weights spanning its range of use (weights that have been calibrated to ASTM Type 1 weights may also be used for daily verification). ASTM Type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every five years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM Type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Reference SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration. A list of balances is in Table 21.2.

21.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

21.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every three years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer has increments of 0.2 °C, and has a range applicable to all method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logsheets. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logsheets. More information on this subject can be found in SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration.

21.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day (seven days a week for DOD labs).

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between or $4 \pm 2^{\circ}\text{C}$.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logsheets posted on each unit and method-specific logbooks.

21.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are checked for accuracy at least quarterly. Glass micro-syringes are considered the same as Class A glassware.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

21.3.6 Field Sampling Devices (ISCO Autosamplers)

Each autosampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is recorded on the sampling documentation in a logbook.

The autosampler is calibrated semi-annually by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The autosampler is programmed to run three cycles, and each of the three cycles is measured into a beaker to verify 100 ml are received.

If the RSD (Relative Standard Deviation) between the three cycles is greater than 20%, the procedure is repeated. If the result is still greater than 20%, the following options may be employed:

- 1) The unit is taken out of service.
- 2) The unit is used to pull composite samples only over a 24-hour period.

The results of this check are kept in a logbook/binder.

21.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 13).

Note: Instruments are calibrated initially and as needed after that and at least annually.

CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. However, the general procedures are described below.

- 21.4.1.1 For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.
- 21.4.1.2 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log is maintained for each department, containing concentration, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.
- 21.4.1.3 The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).
- 21.4.1.4 The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The lowest calibration standard must be at or below the reporting limit. The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.
- 21.4.1.5 Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration will then involve the analysis of each of these sets of the appropriate number of standards.
- 21.4.1.6 All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

21.4.2 CALIBRATION FOR ORGANIC METHODS (GC, HPLC, GC/MS)

- 21.4.2.1 Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multippeak chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. For more details on the calibration types listed below, refer to SOP CA-Q-S-005, Calibration Curves.
- 21.4.2.2 Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are a several EPA methods that have different requirements and are exceptions (e.g. EPA 547) where a minimum of three calibration standards are prepared and analyzed.
- 21.4.2.3 The standard solutions are introduced into the instrument in the same manner as samples are; whether it be by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the five standards.
- External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF).
 - Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution. Not all internal standards need to be at the same concentration in this solution. The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs, e.g., 10 uL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations.

An ideal internal standard concentration would yield a response factor of one for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response, e.g., area counts, that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP CA-Q-S-005, Calibration Curves, for specific calculations.

21.4.2.4 Policies regarding the use of calibration standard results for creating the calibration curve are as follows:

- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.
- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
- Mid-calibration standards may not be excluded unless an obvious reason is found, i.e., cracked vial, incorrectly made, etc. The failed standard should be re-run immediately and inserted into the initial calibration. If not useful, recalibration is required.

21.4.2.5 **Percent RSD Corrective Action**

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

- 21.4.2.5.1** The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other

compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

21.4.2.5.2 If the RSD for any analyte is greater than the applicable acceptance criteria in the applicable analytical method, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

21.4.2.5.3 A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

Note: When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

21.4.2.6 Alternatively, the least squares regression may be used to determine linearity. A five-point line must result in a correlation coefficient (r) of 0.990 or better using the least squares method to be considered acceptable.

21.4.2.7 Instead of a linear curve model (either Average RF or least squares regression), a second order curve (Quadratic) may be used (and preferred) as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or r^2) for the quadratic curve must be at least 0.99 for it to be considered acceptable. For more details on the calculations see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of quadratic curve fits:

21.4.2.7.1 Care **MUST** be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.

21.4.2.7.2 They **may not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).

21.4.2.7.3 They **may not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1st order fit or average RF.

21.4.3 Calibration for Inorganic Analyses

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition) may also be included. Standard Operating Procedures for the analysis and the quality control documentation measures are available electronically on the public drive.

In general, inorganic instrumentation is calibrated with external standards. Some exceptions would be Inductively Coupled Plasma (ICP), Inductively Coupled Plasma Mass Spec (ICPMS), and Ion Chromatography Mass Spec (ICMS). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used. The calibration model in Section 21.4.2.6 is generally used for most methods, however in some instances the model from Section 21.4.2.7 may be used. A correlation coefficient (r) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by hand-held scientific calculators or by computer programs [state what your lab uses] and documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation.

21.4.3.1 "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (20th Edition) for more information.

21.4.3.2 Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.

21.4.3.3 Instrument technologies, e.g., ICP, with validated techniques from the instrument manufacturer or other methods using a zero point and single point calibration require the following:

21.4.3.3.1 The instrument is calibrated using a zero point and a single point calibration standard.

21.4.3.3.2 Sample results within the established linear range do not need to be qualified.

21.4.3.3.3 The linearity is verified at a frequency established by the manufacturer or method.

21.4.4 Calibration Verification

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration

factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration, and is not appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

- 21.4.4.1 Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.
- 21.4.4.2 A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after every 10 samples.
- 21.4.4.3 The acceptance limits for calibration verifications can be found in each method SOP. As a rule of thumb: GCMS $\pm 20\%$, GC and HPLC $\pm 15\%$, Inorganics: ± 10 or 15% . Actual methods may have wider or tighter limits (see the Method SOP for specifics).
- 21.4.4.4 If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.
- 21.4.4.5 If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:
 - 21.4.4.5.1** When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
 - 21.4.4.5.2** When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

21.4.4.6 **Verification of Linear Calibrations**

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(\text{CF(v) or RF(v)}) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

Where: CF(v) or RF(v) = CF or RF from verification standard
Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

21.4.4.7 **Verification of a Non-Linear Calibration**

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations described in Section 21.4.4.6 above.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

21.5 POLICY ON TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it will not be reported as a TIC. If the compound is reported on the same form as true TICs, it must be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See the following SOPs for guidelines for making tentative identifications (CORP-MS-0001NC, GC/MS Analysis Based on Method 8270C, and CORP-MS-0002NC, Determination of Volatile Organics by GC/MS based on Method 8260B & 8260A).

21.6 POLICY ON GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

21.6.1 The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

21.6.2 Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex +/- 1 scan and average the three scans. Background correction is required prior to the

start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

21.6.3 Other Options or if Auto Tune Fails:

- 21.6.3.1 Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak +/- 1 scan and background correct as in 21.6.4 above. This is consistent with EPA 8260 and 8270.
- 21.6.3.2 Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.
- 21.6.3.3 Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as all of the subsequent injections in the 12-hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than two tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in the Instrument Log.
- 21.6.3.4 A single scan at the Apex (only) may also be used for the evaluation of the tune. For SW 846 and EPA 600 series methods, background correction is still required.
- 21.6.3.5 Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. Note: If significant maintenance was performed, see methods 8000B or 8000C then the instrument may require recalibration prior to proceeding.

21.6.4 Tune evaluation printouts must include the chromatogram and spectra as well as the Tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability should be built into the instrument software.

21.6.5 All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.

Table 21-1.

Example - Laboratory Equipment and Instrumentation

Instrument Type	Manufacturer	Model	Auto-sampler
Metals ICP	Thermo Jarrell Ash	Trace Analyzer 61E	Yes
	Thermo Jarrell Ash	Trace Analyzer 61E	Yes
Metals ICP/MS	Perkin Elmer (I-7)	ELAN 6100	Yes
	Thermo (I-8)	Series 2	Yes
Metals Mercury Analyzer	Leeman (CVAA)	PS200 II	Yes
	Leeman (CVAF) Low Level	Hydra AF Gold+, Model #112-00067-1	Yes
	Leeman (CVAA)	Hydra AA	Yes
GC/MS Semivolatiles	Agilent A4AG2	5975-C	NA
	Hewlett-Packard HP7	5973-6890	NA
	Hewlett-Packard HP8	5973-6890	NA
	Hewlett-Packard HP9	5973-6890	NA
	Hewlett-Packard HP10	5973-6890	NA
GC/MS Volatiles	Hewlett-Packard (UX2)	5971A-5890	Yes
	Hewlett-Packard (HP6)	5973-6890	Yes
	Hewlett-Packard (UX7)	5973-6890	Yes
	Hewlett-Packard (UX8)	5973-6890	Yes
	Hewlett-Packard (UX9)	5973-6890	Yes
	Hewlett-Packard (UX10)	5973-6890	Yes
	Agilent (UX11) (former HP)	5973-6890	Yes
	Agilent (UX12)	5973-6890	Yes
	Agilent (UX14)	5973-6890	Yes
	Agilent (UX15)	5973-6890	Yes
	Agilent (UX16)	5975-6890	Yes
GC/MS Volatiles Autosampler	OI Analytical (UX2)	4552	
	OI Analytical (HP6)	4552	
	OI Analytical (UX7)	4552	
	OI Analytical (UX8)	4552	
	OI Analytical (UX9)	4552	
	OI Analytical (UX10)	4552	
	OI Analytical (UX11)	4552	
	OI Analytical (UX12)	4552	
	OI Analytical (UX14)	4552	
	OI Analytical (UX15)	4552	
	OI Analytical (UX16)	4552	

Instrument Type	Manufacturer	Model	Auto-sampler
GC/MS Volatiles Purge and Trap	OI Analytical (UX2)	4560	
	OI Analytical (HP6)	4560	
	OI Analytical (UX7)	4660	
	OI Analytical (UX8)	4560	
	OI Analytical (UX9)	4560	
	OI Analytical (UX10)	4560	
	OI Analytical (UX11)	4560	
	OI Analytical (UX12)	4560	
	OI Analytical (UX14)	4560	
	OI Analytical (UX15)	4660	
	OI Analytical (UX16)	4660	
	EST (spare)	Encon	
WC Ion Chromatograph	Dionex	DX-320	NA
	Dionex	DX-120	NA
WC TOC	OI Analytical	1010 TOC Analyzer	
WC TOX	Thermo Electron	1200-S/N 973515	
	Thermo Electron	1200-S/N 2001.0174	
	Thermo Electron	1200-S/N 2005.0234	
WC UV/VIS	Milton Roy	Spectronic 401	No
	Genesys	Spectronic 20	No
WC Autotitrator	Man-Tech	PC – Titrate	
WC pH Meter	Orion pH Meter	250A	
	Orion (Ammonia ISE)	520A	
WC Dissolved Oxygen Meter	YSI	52C E	No
WC Turbidimeter	HF Scientific	Micro 100	No
WC BOD	Labtronics, Inc.	BOD	
WC Block Digester	Andrews	2210 Phenol	
	Andrews	2205 Ammonia	
	Lachat	BD46 TKN	

Instrument Type	Manufacturer	Model	Auto-sampler
WC Cyanide	Midi Serial #1000-99-01	PRG-2520-BL	
WC Conductivity	Man-Tech	4310	
WC Flashpoint	Petrolab (199443)	Petrotest	
	Herzog	HFP 339	
WC Discrete Analyzer	Kone	Konelab 200	
	Kone	Konelab 250	
WC Residual Chlorine Meter	Hanna	HI 93701	
	Hanna	HI 93701	
WC TRAACS	Bran & Luebbe	800	
Ext. 6-Position Accelerated Soxhlet Extractor	Gerhardt Soxtherm	6 units	
Extractions Sonicators	Misonix	3000	NA
	Fisher	550	NA
GC Semivolatiles	Hewlett-Packard (P1)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P2)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P3)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P4)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P5)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P6)	6890 EPC & Dual FID	Yes
	Hewlett-Packard (P7)	6890 EPC & Dual FID	Yes
	Hewlett-Packard (P9)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P10)	6890 EPC & Dual ECD Y-Splitter	Yes

Instrument Type	Manufacturer	Model	Auto-sampler
GC Semivolatiles (Cont'd)	Hewlett-Packard (P11)	6890 EPC & Dual ECD Y-Splitter	Yes
GC Semivolatiles HPLC	Hewlett-Packard (L2)	HPLC 1100	Yes
	Hewlett-Packard (L3)	HPLC 1100	Yes
GC Volatiles	Agilent (A)	6890 PID/FID	Yes
	Hewlett-Packard (O)	6890 Dual PID/Hall	Yes
	Hewlett-Packard (P)	6890 PID/HALL	Yes
	Hewlett-Packard (Y)	6890N PID/FID	Yes
	Hewlett-Packard (P8)	6890 EPC & Dual FID	Yes
GC Volatiles Auto Sampler	OI Analytical (O)	Archon	
	OI Analytical (Y)	4552	
	Varian (A)	Archon	
	Varian (P)	4552	
GC Volatiles Purge & Trap	Tekmar (O)	4560	
	Tekmar (P)	3000	
	OI Analytical (A)	4560	
	OI Analytical (Y)	3000	
GM Bedford	Agilent	6890N	Yes
	Agilent	6890N	Yes
	Agilent	6890N	Yes
	Misonix Sonicator	3000	NA

Table 21-2.

Example – Laboratory Balance Inventory

Instrument	Location	Manufacturer	Model Number	Type	Serial Number
B001	MS Volatiles	OHAUS	E400D	Top Loader	3317
B002	MS Volatiles	Mettler	AC 100	Analytical	B01177
B005	Metals	Sartorius	R300S	Analytical	38020045
B006	Wet Chemistry	American Scientific	SP180	Analytical	2904794
B007	High Hazard Room	American Scientific	ER-180A	Analytical	2904257
B008	Waste Building (no log book)	Howe Richardson	XL 5401		A029390
B009	Leachates (Extractions)	OHAUS	GT 4800	Top Loader	1687
B010	Wet Chemistry	Mettler	PJ 3600	Top Loader	G29475
B011	Extractions	American Scientific	SP 180	Analytical	2902127
B013	Extractions	OHAUS	TS 4KD	Top Loader	1936
B018	GC Volatiles	American Scientific	DTL 350	Analytical	10594
B019	GC Volatiles	Sartorius	L2200S	Top Loader	36020158
B022	Wet Chemistry	Mettler	PM300	Top Loader	F03040
B023	Wet Chemistry	OHAUS	TS 400S	Top Loader	3608
B024	Wet Chem Prep Room	Mettler	AE 100	Analytical	C25750
B025	Extractions	Mettler	PM 4600	Top Loader	G64548
B027	Wet Chemistry	American Scientific	SP 180	Analytical	2904154
B028	Wet Chemistry	Denver Instrument	M Series	Analytical	P119656
B030	Metals (new 9/5/6)	AND	EK410i	Top Loader	P1841870
B031	Metals (new 9/5/6)	AND	EK410i	Top Loader	P1841872
B032	Metals	OHAUS	ARC120	Top Loader	H28312
B033	Leachates (Extractions)	OHAUS	ARC120	Top Loader	H27612

Table 21-3.

Example – Laboratory Refrigerators, Freezers, Ovens, Thermometers, Steambaths, Hotblocks, and Hotplates

Instrument Type	Manufacturer / Model	Location	Tracking No.
Refrigerators	American Scientific CLP	BNA	R015
	VWR Scientific	BNA	R027
	Frigidaire	BNA	R031
	GE GPC	Extractions	R007
	Hotpoint “D”	Extractions	R008
	GE	Extractions	R010
	Hotpoint CLP Pest.	GC Semi	R016
	VWR	GC Semi	R021
	Frigidaire	GC Semi	R030
	Kelvinator	GC Semi	R032
	BB	GC VOA	R017
	True	MS Voa	R028
	Cryo Frig A CLP	MS Voa	R001
	Mr. Winter	MS Voa	R002
	#203 Enseco	MS Voa	R003
	Hotpoint “OO”	Org Haz	R012
	Kelvinator	Org Prep	R011
	Frigidaire	R&D	R013
	Sample Walk-In 2 nd	Sample Receiving	R018
	LaCross	Sample Receiving	R019
	Walk-In CLP	Sample Receiving	R020
	True	Sample Receiving	R025
	Baxter Cryo	Sample Receiving	R033
	True	Wet Chem	R024
	Frigidaire BOD	Wet Chem	R023
	Baxter Cryo Fridge BOD	Wet Chem	R029
	Frigidaire BOD	Wet Chem	R034
	Fisher Scientific 307 BOD	Wet Chem	R004
Ovens	TDS	Wet Chem	O-001
	WWR	Wet Chem	O-002
	TSS	Wet Chem	O-003
	TDS Evap	Wet Chem	O-004
	Lindberg/Blue	Wet Chem	O-006

Instrument Type	Manufacturer / Model	Location	Tracking No.
Freezers	Kenmore	BNA	F005
	Frigidaire	BNA	F012
	Hotpoint "D"	Extractions	F007
	Magic Chef	GC Semi	F004
	Marvel HPLC	GC Semi	F013
	GE	GC VO	F002
	Kenmore Side/Side	MS Voa	F001
	Frigidaire	MS Voa	F011
	Frigidaire	MS Voa	F016
	Kelvinator	Org Prep	F003
	Crosley	Receiving - Warehouse	F008
	GE	Receiving - Warehouse	F009
	Frigidaire	Receiving - Warehouse	F014
NIST Thermometers	Ertco S/N 69887 Product 15041D		92564
	Ertco S/N NB179010 Product N/A		167513
	Ertco S/N 3053 Product ASTM 068C-BF		15169121
	Ertco S/N 3243 Product ASTM 62C		163757
Steambaths		Extractions	A
		Extractions	B
		Extractions	C
Hotblocks		Metals	0
		Metals	1
		Metals	2
		Metals	3
		Metals	4
		Metals	A1
		Metals	A2
		Metals	B1
		Metals	C1
		Metals	C2
		Metals	C3
		Metals	C4
Hotplate		Metals	1-A
		Metals TCLP	9 K

Table 21-4.

Example: Schedule of Routine Maintenance

(Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations)

**INSTRUMENT MAINTENANCE SCHEDULE
ION CHROMATOGRAPH**

As Needed	Daily	Weekly	Monthly
Clean micro-membrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.
Check fuses when power problems occur.	Check gases.	Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure.		
De-gas pump head when flow is erratic.	Check conductivity meter.		

**INSTRUMENT MAINTENANCE SCHEDULE
TOTAL ORGANIC HALIDE ANALYZER**

Daily	As Needed
Check electrodes for damage; polish the electrodes.	Examine and clean or replace pyrolysis tube.
Replace dehydrating fluid and electrolyte fluid.	Clean titration cell.
Clean quartz boat.	Observe gas flow.
Observe check valves during use for backfeed.	Replace reference electrode fluid.
At end of each day of use, wash out absorption module, empty electrolyte and fill cell with DI water. Empty dehydrator tube	Change quartz wool.
Perform cell performance check.	Replace O-rings and seals.

**INSTRUMENT MAINTENANCE SCHEDULE
HIGH PRESSURE LIQUID CHROMATOGRAPH**

Daily	As Needed
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

**INSTRUMENT MAINTENANCE SCHEDULE
ICP AND ICP/MS**

Daily	Monthly or As Needed	Semi-Annually	Annually
Check vacuum pump gage. (<10 millitorr)	Clean plasma torch assembly to remove accumulated deposits	Change vacuum pump oil	Notify manufacturer service engineer for scheduled preventive maintenance service
Check cooling water supply system is full and drain bottle is not full. Also drain tubing is clear, tight fitting, and has few bends.	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance	Replace coolant water filter (may require more or less frequently depending on quality of water)	
Check nebulizer is not clogged	Clean filters on back of power unit to remove dust		
Check capillary tubing is clean and in good condition	Replace when needed: - peristaltic pump tubing - sample capillary tubing - autosampler sipper probe		
Check peristaltic pump windings are secure	- Check yttrium position - Check O-rings - Clean/lubricate pump rollers		
Check high voltage switch is on			
Check torch, glassware, aerosol injector tube, and bonnet are clean			

INSTRUMENT MAINTENANCE SCHEDULE CVAS AND CVAFS

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading	Lubricate pump	
Check tubing		

INSTRUMENT MAINTENANCE SCHEDULE GAS CHROMATOGRAPH

Daily *	As Needed
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	<p>Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance, or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.</p> <p>Quarterly FID: clean detector, only as needed—not quarterly/or semi-annually.</p>
Check temperatures of injectors and detectors. Verify temperature programs by RT shift.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Clean injector port weekly for TPH for 8015B, when breakdown fails; otherwise, when RT shift or bad samples run.	<p>Annually FID: replace flame tip, only as needed.</p> <p>Only as needed: ECD--detector cleaning and re-foiling, whenever loss of sensitivity, erratic response, or failing resolution is observed</p>
Check baseline level during analysis of run—not maintenance.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).

<p>Watched weekly: check reactor temperature of electrolytic conductivity detector.</p> <p>Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks, when analyzing pesticides; part of analysis—not maintenance.</p> <p>Clip column leader when chromatography looks bad—not daily.</p>	<p>Replace or repair flow controller if constant gas flow cannot be maintained.</p> <p>Replace fuse.</p> <p>Reactivate external carrier gas dryers.</p> <p>Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturer's suggested maintenance schedule.</p> <p>Reactivate flow controller filter dryers when presence of moisture is suspected.</p> <p>HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.</p>
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*No daily maintenance done on any instrument/method. Weekly change IPL on TPH instrument. Everything else is on an "as needed" basis.

**INSTRUMENT MAINTENANCE SCHEDULE
MASS SPECTROMETER**

Daily	Weekly	As Needed	Quarterly	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43)	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between maintenance.	Check ion source and analyzer (clean, replace parts as needed)	Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows	
Check inlets, septa		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump.	
Check baseline level		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates need for replacement.		

**INSTRUMENT MAINTENANCE SCHEDULE
TRAACS AUTO ANALYZER**

As Needed	Daily
Replaces air filter when progressive loss of air pressure is observed.	Check air pressure gauge (22 ± 2 psi)
Replace air valve tubing when occlusion in tubing is observed	Use recommended washout procedure (at end of analysis operations)
Change all pump tubes (or after 200 hours of pumping time, or after 1000 hours of pumping time)	
Clean sample probe shaft	
Replace pump platens	
Lightly lubricate the linear sample rails (use semi-fluid lubricant)	
Replace colorimeter lamp (or after 2500 hours of use)	

**INSTRUMENT MAINTENANCE SCHEDULE
ANALYTICAL/TOP LOADING BALANCES**

Daily	Annually
Check using Class 1-verified weights once daily or before use	Manufacturer cleaning and calibration
Clean pan and weighing compartment	

**INSTRUMENT MAINTENANCE SCHEDULE
REFRIGERATORS/WALK-IN COOLERS**

Daily	As Needed
Temperatures checked and logged	Refrigerant system and electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE
OVENS**

Daily	As Needed
Temperatures checked and logged	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE
SPECIFIC DIGITAL ION ANALYZER**

Daily	As Needed
Daily when used: <ul style="list-style-type: none"> • Calibrate with check standards • Inspect electrode daily, clean as needed • Inspect electrode proper levels of filling solutions daily; fill as needed • Clean probe after each use 	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE
TURBIDIMETER**

Daily	Monthly	As Needed
Daily when used: <ul style="list-style-type: none"> • Adjust linearity on varying levels of NTU standards. Standardize with NTU standards • Inspect cells 	Clean instrument housing	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE
DISSOLVED OXYGEN METER**

Daily	As Needed
Daily when used: <ul style="list-style-type: none"> • Calibrate with saturated air • Check probe membrane for deterioration • Clean and replace membrane with HCl solution 	<ul style="list-style-type: none"> • Electronics serviced • Clean and replace membrane with HCl solution

**INSTRUMENT MAINTENANCE SCHEDULE
CONDUCTANCE METER**

Daily	As Needed
Daily when used: <ul style="list-style-type: none"> • Check probe and cables • Inspect conductivity cell 	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE
CHEMICAL OXYGEN DEMAND (COD) REACTOR ¹**

Daily	As Needed
Daily when used: <ul style="list-style-type: none"> • Calibrate with check standards 	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE
SPECTROPHOTOMETER**

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens	Check the zero % adjustment	Clean windows	Check instrument manual
	Clean sample compartment		Perform wavelength calibration
	Clean cuvettes		Replace lamp annually or when erratic response is observed
			Clean and align optics

INSTRUMENT MAINTENANCE SCHEDULE
pH METER

As Needed	Daily
Clean electrode	Inspect electrode. Verify electrodes are properly connected and filled
Refill reference electrode	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer

INSTRUMENT MAINTENANCE SCHEDULE
TOTAL ORGANIC CARBON ANALYZER

Daily	As Needed	Weekly	Monthly
Check: <ul style="list-style-type: none"> • Oxygen supply • Persulfate supply • Acid supply • Carrier gas flow rate (~ 150 cc/min) • IR millivolts for stability (after 30 min. warm-up) • Reagent reservoirs 	Check injection port septum after 50-200 runs Tube end-fitting connections after 100 hours or use Indicating drying tube NDIR zero, after 100 hours of use Sample pump, after 2000 hours for use Digestion vessel/condensation chamber, after 2000 hours of use Permeation tube, after 2000 hours of use NDIR cell, after 2000 hours of use Change pump tubing	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test

**Instrument Maintenance Schedule
Digestion Block**

Annually
Check temperature with NIST thermometer

**Instrument Maintenance Schedule
Flash Point Tester**

Daily
Check tubing Clean sample cup each use
Check gas
Clean flash assembly
Check stirrer

Table 21-5.

Example: Periodic Equipment Calibrations

Type of Equipment	Calibration Requirements
Balances	<ul style="list-style-type: none"> • Must be serviced and calibrated annually by an approved vendor. • Calibration must be checked daily or before use by analyst with weight(s) classified as Class 1 (formerly termed Class S) by NIST or Class 1 traceable. Acceptance criteria vary according to weight used and accuracy of balance. Acceptance criteria must be documented in the log. • All Class 1 weights must be certified by an outside vendor every three years. • All non-Class 1 weights must be checked annually against NIST Class 1 weights annually.
Thermometers	<ul style="list-style-type: none"> • Working glass thermometers must be calibrated against a certified. • NIST thermometer at least annually as described in operation-specific SOPs. • Working non-glass thermometers must be calibrated against a certified NIST thermometer annually as described in operation-specific SOPs. • The NIST thermometer must be recertified every three years.
Refrigerators/ Freezers	<ul style="list-style-type: none"> • Thermometers must be immersed in a liquid such as mineral oil or glycol. • Temperature of units used for sample or standard storage must be checked daily as described in operation-specific SOPs. Refrigerator acceptance limits: $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Freezer acceptance limits: $< -10^{\circ}\text{C}$
Ovens	<ul style="list-style-type: none"> • Temperature of units must be checked daily or before use. • Acceptance limits vary according to use as described in operation-specific SOPs, and must be documented in the temperature log.
Micropipettes	<ul style="list-style-type: none"> • Calibrations are checked gravimetrically as required by the operation-specific SOP. • Must be calibrated at the frequency (normally quarterly) required by the manufacturer at a minimum.
Syringes, Volumetric Glassware and Graduated Glassware	<ul style="list-style-type: none"> • All syringes and volumetric glassware are purchased as Class A items. • Class A items are certified by the manufacturer to be within $\pm 1\%$ of the measured volume; therefore, calibration of these items by TestAmerica laboratories is not required. • All analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents, and sample volumes are within method tolerances.

Table 21-6.

Example: Preventive Maintenance Procedures

SUMMARY OF INORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Acidity	Initial	350.1	Two-level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	350.1	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	350.1	Third point check	9040B 9045C	Third point check
	Ending	350.1	One buffer check ± 5% of true value	9040B 9045C	N/A
Alkalinity, Bicarbonate, Carbonate	Initial	310.1 2320B	2 point calibration of pH meter ± 0.05 pH units of true value	--	N/A
	Continuing	310.1 2320B	N/A	--	N/A
	Ending	310.1 2320B	N/A	--	N/A
Ammonia	Initial	350.1	6 levels including blank, "r" ³ ≥ 0.995	--	N/A
	Continuing	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
	Ending	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
Arsenic Speciation		N/A	N/A	7063	* Refer to Section 10 of SOP NC-WC-0090

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Biochemical Oxygen Demand (BOD)	Initial	405.1 SM5210B	a. Winkler titration: Iodometric with standard thiosulfate b. Membrane electrode: Read in air and in water with zero dissolved oxygen	--	N/A
	Continuing	405.1 SM5210B	N/A	--	N/A
	Ending	405.1 SM5210B	N/A	--	N/A
Bromide	Initial	300.0A	5 levels plus a blank, " $r^3 \geq 0.995$ "	9056A	5 levels plus a blank, " $r^3 \geq 0.995$ "
	Continuing	300.0A	Level every 10 samples $\pm 10\%$ of true value	9056A	N/A
	Ending	300.0A	N/A	9056A	N/A
Cation Exchange	Initial	N/A	N/A	9081	N/A
	Continuing	N/A	N/A	9081	N/A
	Ending	N/A	N/A	9081	N/A
Chemical Oxygen Demand (COD)	Initial	410.4 SM5220D	5 levels plus a blank " $r^3 \geq 0.995$ "	--	N/A
	Continuing	410.4 SM5220D	One level every 10 samples $\pm 10\%$ of true value	--	N/A
	Ending	410.4 SM5220D	One level $\pm 10\%$ of true value	--	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Chloride	Initial	300.0A 325.2	5 levels plus blank $"r" \geq 0.995$	9056A 9251	<u>Method 9056:</u> 3 levels plus a blank <u>Method 9252:</u> 5 levels plus blank $"r" \geq 0.995$
	Continuing	300.0A 325.2	One level every 10 samples $\pm 10\%$ of true value	9056A 9251	<u>Method 9056:</u> One per batch of 20 samples, $\pm 10\%$ of true value <u>Method 9252:</u> One level every 10 samples, $\pm 10\%$ of true value
	Ending	300.0A 325.2	One level every 10 samples $\pm 10\%$ of true value	9056A 9251	<u>Method 9056 :</u> N/A <u>Method 9252:</u> One level $\pm 10\%$ of true value
Chromium Cr ⁺⁶	Initial	3500 Cr-D	3 levels plus blank	7196A	5 levels plus blank $"r" \geq 0.995$
	Continuing	3500 Cr-D	One level every 10 samples $\pm 10\%$ of true value	7196A	One level every 10 samples $\pm 15\%$
	Ending	3500 Cr-D	One level $\pm 10\%$ of true value	7196A	One level $\pm 15\%$
Chlorine, Residual	Initial	330.5 SM3500CL-G	N/A	--	N/A
	Continuing	330.5 SM3500CL-G	N/A	--	N/A
	Ending	330.5 SM3500CL-G	N/A	--	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Conductivity	Initial	120.1 SM2510B	Standard KCl solution	9050A	One level to determine cell constant
	Continuing	120.1 SM2510B	N/A	9050A	N/A
	Ending	120.1 SM2510B	N/A	9050A	N/A
Cyanide (Amenable)	Initial	335.1 SM4500CN-G	6 levels plus blank "r" ³ ≥ 0.995	9012A	6 levels plus blank "r" ³ ≥ 0.995
	Continuing	335.1 SM4500CN-G	One level every 10 samples ± 10% of true	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1 SM4500CN-G	One level ± 10 % of true value	9012A	± 15% of true value
Cyanide (Total)	Initial	335.1/335.2 335.3/335.4 SM4500CU-E	6 levels plus blank "r" ³ ≥ 0.995	9012A	6 levels plus blank "r" ³ ≥ 0.995
	Continuing	335.1/335.2 335.3/335.4 SM4500CU-E	One mid-level every 10 samples ± 10 % of true value	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1/335.2 335.3/335.4 SM4500CU-E	One mid-level ± 10 % of true value	9012A	± 15% of true value
Flashpoint	Initial	--	N/A	1010 ASTM D93-9	p-Xylene reference standard must have flashpoint of 81°F ±2°F
	Continuing	--	N/A	1010 ASTM D93-9	N/A
	Ending	--	N/A	1010 ASTM D93-9	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Fluoride	Initial	300.0A 340.2	Method 300.0A: 5 levels plus a blank, "r" ³ ≥ 0.995 Method 340.2: 5 levels "r" ³ ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 340.2	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 340.2	One mid-level ± 10% of true value	9056A	N/A
Hardness	Initial	130.2 2340B	Method 130.2: Standardize titrant Method 2340B: See ICP Metals 200.7	--	N/A
	Continuing	130.2 2340B	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
	Ending	130.2 2340B	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
Iron (Ferrous)	Initial	3500-Fe D	3 levels plus a blank, "r" ³ ≥ 0.995	-	N/A
	Continuing	3500-Fe D	One mid-level every 10 samples ± 10% of true value	-	N/A
	Ending	3500-Fe D	One mid-level ± 10% of true value	-	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Nitrate	Initial	300.0A 353.2 SM4500NO ₃ -E	5 levels plus a blank "r" ³ ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 353.2 SM4500NO ₃ -E	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 353.2 SM4500NO ₃ -E	One mid-level ± 10% of true value	9056A	N/A
Nitrite	Initial	300.0A 354.1	5 levels plus a blank "r" ³ ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 354.1	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 354.1	One mid-level ± 10% of true value	9056A	N/A
Nitrate-Nitrite	Initial	300.0A 353.2	5 levels plus blank "r" ³ ≥ 0.995	--	N/A
	Continuing	300.0A 353.2	One level every 10 samples ± 10% of true value	--	N/A
	Ending	300.0A 353.2	One mid-level ± 10% of true value	--	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Phosphorus (Total and Ortho-phosphate)	Initial	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: 3 levels plus a blank Method 365.2: 5 levels plus a blank	--	N/A
	Continuing	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: One level every 10 samples ± 10% of true value Method 365.2: Blank and 2 standards with each series of samples, ± 2% of true value or recalibrate	--	N/A
	Ending	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: ± 10% of true value Method 365.2: N/A	--	N/A
pH	Initial	150.1 SM4500H-B	2 level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	150.1 SM4500H-B	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	150.1 SM4500H-B	Third point check	9040B 9045C	Third point check
	Ending	150.1 SM4500H-B	One buffer check ± 5% of true value	9040B 9045C	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Phenolics	Initial	420.1	5 levels plus a blank "r" ³ ≥ 0.995	9065 9066	5 levels plus a blank "r" ³ ≥ 0.995
	Continuing	420.1	One mid-level every 10 samples ± 10% true value	9065 9066	One mid-level ± 15% true value
	Ending	420.1	One mid-level ± 10% true value	9065 9066	One mid-level ± 15% true value
Phosphate	Initial	SM4500P-E	N/A	9056	3 levels plus a blank
	Continuing	SM4500P-E	N/A	9056	One per batch of 20 samples ± 15% of true value
	Ending	SM4500P-E	N/A	9056	N/A
Settleable Solids	Initial	160.5	N/A	--	N/A
	Continuing	160.5	N/A	--	N/A
	Ending	160.5	N/A	--	N/A
Specific Conductance	Initial	120.1 SM2510B	Standardize meter with 0.01 M KCl	9050A	N/A
	Continuing	120.1 SM2510B	One level every 10 samples ± 10% of true value	9050A	N/A
	Ending	120.1 SM2510B	One level ± 10% of true value	9050A	N/A
Sulfate	Initial	300.0A 375.4	<u>Method 300.0A:</u> 5 levels plus blank "r" ³ ≥ 0.995 <u>Method 375.4:</u> 3 levels plus blank "r" ³ ≥ 0.995	9038 9056A	<u>Method 9038:</u> 3 levels plus a blank for every hour of continuous sample analysis. <u>Method 9056:</u> 3 levels plus a blank

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Sulfate (Cont'd)	Continuing	300.0A 375.4	<u>Method 300.0A:</u> One mid-level after every 10 samples ± 10% of true value <u>Method 375.4:</u> One level every 3 or 4 samples ± 10% of true value	9038 9056A	<u>Method 9038:</u> Independent-prepared check standard every 15 samples <u>Method 9056:</u> 1 per batch of 20 samples ± 10% of true value
	Ending	300.0A 375.4	± 10% of true value	9038 9056A	N/A
Sulfide	Initial	376.1	<u>Method 376.1:</u> This is a titration method. Therefore, calibrations are not applicable.	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Continuing	376.1	<u>Method 376.1:</u> N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Ending	376.1	<u>Method 376.1:</u> N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
Total Dissolved Solids	Initial	160.1 SM2540E	This is a gravimetric determination. Calibrate balance prior to analysis	--	N/A
	Continuing	160.1 SM2540E		--	N/A
	Ending	160.1 SM2540E		--	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Total Kjeldahl Nitrogen (TKN)	Initial	351.3 SM4500NO ₃	Method 351.3: Titrimetric: Standardize titrant Colorimetric: 7 levels plus blank	--	N/A
	Continuing	351.3 SM4500NO ₃	Method 351.3: N/A	--	N/A
	Ending	351.3 SM4500NO ₃	Method 351.3: N/A	--	N/A
Total Organic Carbon (TOC)	Initial	415.1 SM5310D	3 levels plus blank	9060 Walkley Black	3 levels plus blank "r" ³ ≥ 0.995
	Continuing	415.1 SM5310D	1 mid-level every 10 samples ± 15% of true value	9060 Walkley Black	1 mid-level every 10 samples ± 15% of true value
	Ending	415.1 SM5310D	± 15% of true value	9060 Walkley Black	± 15% of true value
Total Organic Halides (TOX)	Initial	450.1	Method 450.1: Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently- prepared check standard	9020B 9023 (EOX)	Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently-prepared check standard –ICV ± 10% SOP CORP-WC- 0001

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Total Organic Halides (TOX) (cont'd)	Continuing	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
	Ending	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
Total Solids	Initial	160.3	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.3		--	N/A
	Ending	160.3		--	N/A
Total Suspended Solids (Nonfilterable)	Initial	160.2 SM2540E	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.2 SM2540E		--	N/A
	Ending	160.2 SM2540E		--	N/A
Turbidity	Initial	180.1	Minimum of 1 level in each instrument range. Follow manufacturer's instructions	--	N/A
	Continuing	180.1	N/A	--	N/A
	Ending	180.1	N/A	--	N/A
Volatile Solids	Initial	160.4	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.4		--	N/A
	Ending	160.4		--	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
ICP & ICP/MS Metals (excludes Hg)	Initial	200.7	One level and blank. ICV RSD <3% from replicate	6010B	One level and blank. ICV RSD <5% from replicate
	Initial	200.8	One level and blank	6020	One level and blank
	Continuing	200.7	Every 10 samples ±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD < 5% from replicate
	Continuing	200.8	N/A	6020	N/A
	Ending	200.7	±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard ± 10% of true value CCV RSD < 5% from replicate
	Ending	200.8	N/A	6020	N/A
	Other	200.7	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits	6010B	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits
	Other	200.8	N/A	6020	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Mercury by CVAA & CVAFS	Initial	245.1 1631E	5 levels plus blank ICV $\pm 10\%$ of true value " r " ³ ≥ 0.995	7470A 7471A	5 levels plus blank ICV $\pm 10\%$ of true value " r " ³ ≥ 0.995
	Continuing	245.1* 1631E	Daily or every 10 samples, whichever is more frequent $\pm 20\%$ of true value	7470A 7471A	Every 10 samples $\pm 20\%$ of true value
	Ending	245.1 1631E	$\pm 20\%$ of true value	7470A 7471A	$\pm 20\%$ of original prepared standard
	Other	245.1 1631E	<u>Annually:</u> MDL	7470A 7471A	<u>Annually:</u> MDL

* 245.1 continuing – Initial CCV $\pm 5\%$ of true value

Footnotes

¹ National Pollutant Discharge Elimination System.

² Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December, 1996).

³ " r " = correlation coefficient.

SUMMARY OF ORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Aromatic Volatiles by GC	Initial	602	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	602	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections or 12 hrs whichever is greater, % D ≤ 15%, gases 20% D. Evaluate per SOP
	Ending	602	Run closer per NELAC requirement, but no criteria	8021B	Mid-level calibration standard, % D ≤ 15%. Evaluate per SOP, except as noted above.
	Other	602	N/A	8021B	N/A
Herbicides by GC	Initial	615 ⁹	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8151A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	615 ⁹	One or more calibration standards analyzed daily % D ± 15% of predicted response	8151A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	615 ⁹	N/A	8151A	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	615 ⁹	N/A	8151A	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Pesticides/ PCBs by GC	Initial	608	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8081A 8082	Minimum of 5 levels. If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP CORP-GC-0001)
	Continuing	608	One or more calibration standards analyzed daily. % D \pm 15% of predicted response	8081A 8082	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	608	N/A	8081A 8082	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	608	N/A	8081A 8082	N/A
Petroleum Hydrocarbons/ Oil and Grease	Initial	1664A	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be \pm 10% at 2 mg and \pm 0.5% at 1000 mg or recalibrate balance	9071B	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be \pm 10% at 2 mg and \pm 0.5% at 1000 mg or recalibrate balance
	Continuing	1664A	N/A	9071B	N/A
	Ending	1664A	N/A	9071B	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Purgeable Halocarbons by GC	Initial	601	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	601	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections, or 12 hrs, whichever is greater. % D < 15%, gases 20% D Evaluate per SOP
	Ending	601	Run per NELAC, but no requirement to meet	8021B	Mid-level calibration standard, % D <15%. Evaluate per SOP
	Other	601	N/A	8021B	N/A
Halogenated Volatiles by GC	Initial	--	N/A	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	--	N/A	8021B	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	--	N/A	8021B	Mid-level calibration standard % D < 15% of predicted response for any analyte quantitated and reported.
	Other	--	N/A	8021B	N/A

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Semivolatiles	Initial	625	Minimum of 3 levels, lowest near but above MDL. If % RSD \leq 35%, use avg RF. Otherwise calibration curve employed.	8270C	Minimum of 5 levels, % RSD for RF for CCCs ⁽⁴⁾ < 30% SPCCs ⁽⁵⁾ : RF > 0.050
	Continuing	625	One level every 24 ours. Acceptance criteria are found in the method and SOP.	8270C	Mid-level standard every 12 hours (after tuning) %D for CCCs ⁽⁴⁾ < 20 % between RF from standard and avg RF from initial SPCCs ⁽⁵⁾ : RF > 0.050.
	Ending	625	N/A	8270C	N/A
	Other	625	DFTPP ⁽⁷⁾ tuning every 24 hours before standard or sample runs.	8270C	DFTPP ⁽⁷⁾ tuning at the beginning of every 12 hour shift.

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Volatiles	Initial	624	Minimum of 3 levels, lowest near but above MDL. If % RSD \leq 35%, use avg RF. Otherwise, calibration curve employed.	8260B	Minimum of 5 levels, %RSD for RF for CCCs ⁴ < 30.0% SPCCs ⁵ : RF \geq 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Continuing	624	1 level every 24 hours Acceptance criteria are found in the method and SOP	8260B	Mid-level standard every 12 hours (after tuning) %Drift for CCCs ⁽⁴⁾ < 20.0% between RF from standard and avg RF from initial SPCCs ⁽⁵⁾ : RF \geq 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Ending	624	N/A	8260B	N/A
	Other	624	BFB ⁽⁶⁾ tuning at the beginning of every 24 hour shift.	8260B	BFB ⁽⁶⁾ tuning at the beginning of every 12 hour shift.

Footnotes:

- ¹ National Pollutant Discharge Elimination System.
- ² Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- ³ TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin.
- ⁴ CCC - Continuing Calibration Compounds.
- ⁵ SPCC - System Performance Check Compound.
- ⁶ BFB – Bromofluorobenzene.
- ⁷ DFTPP – Decafluorotriphenylphosphine.
- ⁸ Footnote deleted.
- ⁹ Method not listed in 40 CFR Part 136.

SECTION 22

MEASUREMENT TRACEABILITY (NELAC 5.5.6)

22.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes are verified at least semi-annually or disposed of after six months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. The following definitions are provided by the American Association for Laboratory Accreditation (A2LA):

“Traceability is the property of a measurement result whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, each step in the chain having stated uncertainties.” There are six essential elements:

- An unbroken chain of comparison
- A calculated measurement uncertainty for each step in the chain to allow for an overall uncertainty calculation
- Documentation of each step in each calibration report
- All steps in the chain are performed by individuals with evidence of technical competence and accredited by a recognized accreditation body
- Reference to International Standard (SI) units
- Recalibration at appropriate intervals to preserve traceability

Calibration is defined as “determining and documenting the deviation of the indication of a measuring instrument (or the stated value of a material measure) from the conventional ‘true’ value of the measurand.”

Uncertainty is defined as “a parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measurand.” Measurement of Uncertainty is discussed in Section 20 of this QA Manual.

22.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

22.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional information on purchasing). The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to Table 9-1 in Section 9 for general storage requirements and Table 22-1 for additional storage information. Please refer to SOP NC-QA-0017, Standards and Reagents, for additional details. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

22.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in each group. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

22.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into standard or reagent logbooks, and are assigned a unique identification number. The following information is typically recorded in the Standards Logbook.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation date
- Expiration date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent standard analyte concentration (if applicable)
- Parent standard amount used (if applicable)
- Component analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained in each group for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

22.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration date
- Standard ID (from the Standards Logbook)
- Special health/safety warnings if applicable

22.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority:

- 1) With the manufacturer's recommendations
- 2) With requirements in the specific analytical methods
- 3) According to Table 22-1

SECTION 23.0

SAMPLING (NELAC 5.5.7)

23.1 OVERVIEW

TestAmerica North Canton provides sampling services. Sampling procedures are described in SOP NC-SC-0006, Sample Procurement Protocol.

23.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are available from the vendor electronically, or stored at the laboratory.

23.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

23.2.2 Preparing Container Orders

Container orders are prepared using the laboratory Shipping Department, SOP NC-QA-0012. The laboratory also provides EnCore, TerraCore or other soil sampling devices when requested.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

23.3 FIELD QUALITY CONTROL (QC)

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample

containers for all volatile organic analyses. Blanks generated in the field will be analyzed along with the field samples (exception soil samples where the blank is aqueous).

23.3.1 Equipment Blank / Rinsate Blank - The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water should be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and will also be affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank should begin when the equipment is rinsed and the water is collected. Equipment blank collection for low level mercury is generally done at a clean facility rather than at the sample collection site.

23.3.2 Field Blank - The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank should be when the blank is prepared in the field.

23.3.3 Trip Blank - The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which will be used for sampling. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the "Trip" ends).

23.3.4 Field Duplicates - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

23.4 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g. 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

23.4.1 Semi-Volatile - Holding times for sample preparation for semi-volatile organics are measured from the sampling date (and time where applicable) until the day (and time where applicable) solvent contacts the sample. If a sample is to be extracted on the day of expiration, the actual time of extraction must be recorded on the sample preparation worksheet. Holding

times for analysis are measured from the date (and time where applicable) of initiation of extraction to the time of injection into the gas chromatograph.

23.4.2 Volatiles - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date and time of injection into the gas chromatograph.

23.4.3 Inorganics - For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable).

23.5 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times (refer to Tables 23-1 to 23-7) or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

23.6 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis. In that regard the following guidelines apply to analysts:

Analysts must handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

23.6.1 For water samples, before taking each aliquot for analysis, invert the sample container end-over-end three times and immediately pour off the aliquot. Especially when suspended solids are present, adequate mixing of the sample is extremely important.

23.6.2 For solid samples, when volatile organics are not requested, if the solid can be mixed, stir before removing the aliquot. When possible stir the entire sample in the sample container. Remove small increments of sample from different parts of the container to make up the complete subsample.

- For soil samples, avoid debris in the subsample aliquot as much as possible (e.g. gravel, sticks, roots and grass); note this information in the sample preparation record.

23.6.3 For solid samples, when volatile organics analysis is requested, the sample should be manipulated as little as possible. In most cases, the sample will arrive already preserved or in an EnCore™ sampler of the correct mass (requiring quick preservation of the entire amount). If the client requests volatiles on a solid sample which has been collected in a jar and is in a common container from which aliquots for other test methods must be taken, login should deliver the container to the volatiles department for preparing a proper aliquot prior to any other aliquots being taken out.

23.6.4 For multiphasic samples, the client should instruct the laboratory as to the intent of the testing and how to handle the sample. If the entire sample is to be accounted for, and the phases do not mix easily with inversion/stirring, such that a representative aliquot can be taken, the analyst must record the percent by volume of each phase. The analysis must be conducted on each phase separately; the final results are combined mathematically, weighting the individual phase results by volume. One exception to this procedure is the situation addressed in the TCLP and SPLP methods for wastes containing free liquids. However, if the leachate and final filtrate are not miscible, it is necessary to combine mathematically the concentrations of the two (or more) solutions by volume. A laboratory coliwasa can be used to subsample a multilayer liquid sample when appropriate.

Tables 23-1 to 23-7 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. The sample volumes are intended to be a minimal amount to perform the method. The containers used may be of larger size. **Please note:** The holding times are program specific and different programs may have different holding times for equivalent methods, e.g., there are differences in holding times for many organic analytes between SDWA and NPDES. RCRA methods may also be different.

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3, 7}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method	Requirements
Acidity	Water	100 mL	305.1 SM2310B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
Alkalinity, Bicarbonate, Carbonate	Water	100 mL	310.1 2320B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ammonia	Water	100 mL	350.1 350.2 SM4500NH ₃ -E SM4500NH ₃ -F	500 mL plastic or glass. Cool to 4°C H ₂ SO ₄ to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Arsenic (ASV) Anodic Stripping Voltammetry	Water	100 mL	---	N/A	7063	250 mL plastic. Cool to 4°C. HCl to pH < 2, 28 days
Biochemical Oxygen Demand (BOD), Carbonaceous	Water	1000 mL	405.1 5210B	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Bromide	Water	50 mL	300.0A ⁷	250 mL plastic or glass. No preservative required, 28 days	9056A	Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Cation Exchange	Solid	8 oz	---	N/A	9081	8 or 16 oz glass. Cool to 4°C, 6 months
Chemical Oxygen Demand (COD)	Water	100 mL	410.4 5220D	250 mL glass or plastic. Cool to 4°C, H ₂ SO ₄ to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3, 7}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method	Requirements
Chloride	Water	50 mL	300.0A ⁷ 325.2	250 mL plastic or glass. No preservative required, 28 days	9056A 9251	Method 9056: Cool to 4°C. Analyze ASAP after collection. Method 9251: 250ml plastic or glass, no preservative required, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chlorine, Residual	Water	100 mL	330.5	250 mL glass or plastic. Cool to 4°C, analyze immediately	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chromium (Cr ⁺⁶)	Water	100 mL	3500 Cr-D	Method 218.4: 200 mL plastic or glass. Cool to 4°C, 24 hours. Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO ₃ to pH <2. Cool to 4°C. Analyze ASAP after collection	7196A	200 mL plastic or glass. Cool to 4°C, 24 hours
	Solid	20 g	---	N/A	7196A 3060A	250 mL plastic or glass, 30 days to digestion, 96 hours after digestion
	Waste	N/A	---	N/A	---	N/A
Conductivity	Water	100 mL	120.1 2510B	200 mL glass or plastic. Cool to 4°C, 28 days	9050A	200 mL glass or plastic. Cool to 4°C, 24 hours
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3, 7}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method	Requirements
Cyanide (Amenable)	Water	250 mL	335.1 SM4500CN-G	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid ⁶ . Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid ⁶ . Cool to 4°C, 14 days
	Solid	50g	---	N/A	9012A	Not Specified
	Waste	50g	---	N/A	9012A	Not Specified
Cyanide (Total)	Water	1L	335.2 335.3 335.4 ⁽⁷⁾ SM4500CN-E	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid ⁶ . Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid ⁶ . Cool to 4°C, 14 days.
	Solid	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C, 14 days
	Waste	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C
Flashpoint (Ignitability)	Liquid	100 mL	---	N/A	1010 ASTM D93-9	No requirements, 250 mL amber glass. Cool to 4°C recommended
	Solid	100 g	--	N/A	---	N/A
	Waste	100 mL	--	N/A	---	N/A
Fluoride	Water	300 mL	300.0 ⁷ 340.2	500 mL plastic. No preservation required, 28 days.	9056A	Cool to 4°C. Analyze ASAP after collection.
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3, 7}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method	Requirements
Hardness (Total)	Water	50 mL	130.2 2340B	250 mL glass or plastic, HNO ₃ to pH < 2, 6 months	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Iron (Ferrous)	Water	100 mL	3500-Fe D	1 liter glass or polyethylene container, 6 months. This test should be performed in the field.	-	N/A
	Solid	N/A	-	N/A	-	N/A
	Waste	N/A	-	N/A	-	N/A
Nitrate	Water	50 mL	300.0A ⁷ SM4500NO ₃ -E	Method 300.0: 250 mL plastic or glass. Cool to 4°C, 48 hours.	9056A	Method 9056: Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	Not Specified
Nitrite	Water	50 mL	300.0A ⁷ 354.1	250 mL plastic or glass. Cool, 4°C, 48 hours	9056A	Cool, 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Nitrate-Nitrite	Water	50 mL	353.2	250 mL plastic or glass, H ₂ SO ₄ to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ortho-phosphate	Water	50 mL	300.0A ⁷ 365.1 SM4500P-E	100 mL plastic or glass. Filter on site. Cool to 4°C, 48 hours	9056A	Cool to 4°C. Analyze ASAP collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3, 7}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method	Requirements
pH	Water	50 mL	150.1 SM4500H-B	100 mL plastic or glass. Analyze immediately. This test should be performed in the field.	9040B	100 mL plastic or glass. Analyze immediately. This test should be performed in the field. ⁽⁸⁾
	Solid	N/A	---	N/A	9045C	4 oz glass or plastic. Cool to 4°C. Analyze as soon as possible. ⁸
	Waste	N/A	---	N/A	9045C	4 oz glass or plastic, Cool to 4°C. Analyze as soon as possible. ⁸
Phenolics	Water	100 mL	420.1	500 mL glass, Cool to 4°C, H ₂ SO ₄ to pH < 2, 28 days	9065	1 liter glass recommended, Cool to 4°C, H ₂ SO ₄ to pH < 4, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	9065	Not Specified
Phosphate	Water	50 mL	---	N/A	9056A	Cool to 4°C, analyze ASAP collection
	Solid	N/A	---	N/A	9056A	N/A
	Waste	N/A	---	N/A	9056A	N/A

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3, 7}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method	Requirements
Phosphorus (Total)	Water	100 mL	365.2 365.1 SM4500P-E	100 mL plastic or glass, H ₂ SO ₄ to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Settleable Solids	Water	1000 mL	160.5	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Specific Conductance	Water	50 mL	120.1 2510B	250 mL plastic or glass. Cool to 4°C, 24 hours	9050A	250 mL plastic or glass. Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Sulfate (SO ₄)	Water	50 mL	300.0A ⁷ 375.4	100 mL plastic or glass. Cool to 4°C, 28 days	9056A 9038	Method 9056: Cool to 4°C. Analyze ASAP collection. Method 9038: 200 mL plastic or glass, Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	100 mL	---	N/A	9038	200 mL plastic or glass. Cool to 4°C, 28 days

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3, 7}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method	Requirements
Sulfide	Water	250 mL	376.1	500 mL plastic or glass. Cool to 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030A 9030B/ 9034	500 mL plastic, No headspace. Cool to 4°C. Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days
	Solid	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
	Waste	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
Total Dissolved Solids (Filterable)	Water	100 mL	160.1 2540C	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3, 7}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method	Requirements
Total Kjeldahl Nitrogen (TKN)	Water	100 mL	351.3	500 mL plastic or glass. Cool to 4°C, H ₂ SO ₄ to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Organic Carbon (TOC)	Water	100 mL	415.1 SM5310D	100 mL plastic or glass. Cool to 4°C, H ₂ SO ₄ to pH < 2, 28 days	9060 Walkley-Black	100 mL glass or 40 mL VOA vials, Cool to 4°C, H ₂ SO ₄ or HCl to pH < 2, 28 days
	Solid	N/A	---	N/A	9060 Walkley-Black	Not Specified
	Waste	N/A	---	N/A	9060 Walkley-Black	Not Specified
Total Organic Halides (TOX) (EOX)	Water	100 mL	450.1 ⁽⁷⁾	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, HNO ₃ to pH < 2, no headspace, 28 days	9020B 9023 (EOX)	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, H ₂ SO ₄ to pH < 2, no headspace, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Solids	Water	100 mL	160.3	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Suspended Solids (Nonfilterable)	Water	100 mL	160.2	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3, 7}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method	Requirements
Turbidity	Water	50 mL	180.1	250 mL plastic or glass. Cool, 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Volatile Solids	Water	100 mL	160.4	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO ₃ to pH ≤ 2, 6 months	6010B 6020	1 liter glass or polyethylene container, HNO ₃ to pH ≤ 2, 6 months
	Solid	200 g	200 series	2, 8, or 16 oz glass or polyethylene container storage at 4 °C	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	N/A	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
Mercury (CVAA) (CVAFS)	Water	100 mL	245.1 245.7 1631E	250 mL glass or polyethylene container, HNO ₃ to pH ≤ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO ₃ to pH ≤ 2, 28 days
	Solid	200 g	--	2, 8, or 16 oz glass or polyethylene container. Cool to 4°C, 28 days. Not applicable for Method 1631E.	7471A	8 or 16 oz glass or polyethylene container. Cool to 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	--	N/A	7471A	8 or 16 oz glass or polyethylene container. Cool, 4°C, 28 days (CORP-MT-0007)

Footnotes

- ¹ Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
- ² National Pollutant Discharge Elimination System - MCAWW, March 1983.
- ³ Holding times are calculated from date of collection.
- ⁴ Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- ⁵ Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.
- ⁶ Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.
- ⁷ Method not listed in 40 CFR Part 136.
- ⁸ If not done in the field (ASAP) per the method and requested by client, analyze in lab within 48 hours.
- ⁹ EPA issued memo recommending not to use reactive cyanide and sulfide methods.

Table 23-2.

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method ⁶	Requirements
Aromatic Volatiles	Water	40 mL	602	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 7 days with pH > 2, 14 days with pH ≤ 2	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2
	Solid ⁵	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C ¹²
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Max holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool to 4°C ¹²

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method ⁶	Requirements
Halogenated Volatiles by GC	Water	40 mL	--	N/A	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH ≤ 2, 14 days
	Solid ⁵	5 g or 25 g	--		8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool to 4°C ¹²
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C ¹² .

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method ⁶	Requirements
Herbicides	Water	1L	615 ⁽¹⁰⁾	1 liter amber glass with Teflon®-lined lid, Sodium thiosulfate or ascorbic acid if residual chlorine present. Cool to 4°C, Extraction, 7 days. Analysis, 40 days after extraction	8151A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool to 4°C. Extraction, 7 days. Analysis, 40 days of the start of extraction.
	Solid	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method ⁶	Requirements
Pesticides/ PCBs	Water	1L	608	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C. Extraction, 7 days. Analysis, 40 days after extraction.	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, 40 days of the start of the extraction.
	Solid	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method ⁶	Requirements
Petroleum Hydrocarbons /Oil and Grease	Water	1 L	1664A ⁽⁷⁾	1 liter glass, Cool, 4°C HCl or H ₂ SO ₄ to pH <2 28 days	9071B	1 liter glass, Cool, 0-4°C HCl or H ₂ SO ₄ to pH <2 28 days
	Solid	30 g	1664A ⁽⁷⁾	8 or 16 oz. Wide mouth glass jar, Cool, 4°C, 28 days	9071B	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days
	Waste	---	---	N/A	9071B	N/A
Purgeable Halocarbons by GC	Water	40 mL	601	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 14 days.	8021B	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 1:1 HCl to pH ≤ 2, 14 days.
	Solid	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool, 4°C ¹² .

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method ⁶	Requirements
Purgeable Halocarbons by GC (cont'd)	Waste	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for EnCore™ sampler is 48 hours (before the sample is added to methanol). Cool, 4°C ¹²
Semivolatiles	Water	1L	625	1 liter amber glass with Teflon®-lined lid. Cool, 4°C. Extraction, 7 days. Analysis, 40 days.	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, within 40 days of extraction.
	Solid	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.
	Waste	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.

Analytical Parameters	Matrix	Minimum Sample Size ¹	NPDES ^{2, 3}		RCRA (SW846) ^{3, 4}	
			Method	Requirements	Method ⁶	Requirements
Volatile Organics	Water	40 mL	624	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2 ⁸ .	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2 ⁹ .
	Solid ⁵	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol or sodium bisulfate). Cool to 4°C ⁽¹²⁾
	Waste	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before sample is added to methanol or sodium bisulfate). Cool to 4°C ¹²

Footnotes

- ¹ Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
- ² National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.
- ³ Holding times are calculated from the date of collection.
- ⁴ Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- ⁵ Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.
- ⁶ Only one determination method is listed when separate methods are required for preparation and analysis.
- ⁷ Method 1664 was promulgated by the EPA with an effective date of June 14, 1999.
- ⁸ For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.
- ⁹ For acrolein and acrylonitrile the pH should be adjusted to 4-5.
- ¹⁰ Method not listed in 40 CFR Part 136.
- ¹¹ Should only be used in the presence of residual chlorine.
- ¹² Depending on regulatory programs, EnCore™ samplers may be preserved for up to 14 days from sampling by freezing at -5 to -12°C until analysis. Alternatively the EnCore™ sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon®-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)

Table 23-3.

Sample Containers, Preservatives, and Holding Times for TCLP¹ and SPLP²

Analytical Parameters	Matrix	Minimum Sample Size	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

Footnotes

¹ TCLP = Toxicity Characteristic Leaching Procedure

² SPLP = Synthetic Precipitation Leaching Procedure

SECTION 24

HANDLING OF SAMPLES (NELAC 5.5.8)

Sample management procedures at TestAmerica North Canton ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

24.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and can be initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 24-1.

24.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 24-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The COC is stored with project information and the report.

24.1.2 Legal / Evidentiary Chain-of-Custody

The lab does not accept samples that require legal chain-of-custody.

24.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections. SOP NC-SC-0005, Sample Receiving and Sample Control, describes the laboratory's sample receipt procedure.

24.2.1 Laboratory Receipt

Samples shall be received and logged in at TestAmerica by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of cut-resistant gloves, a lab coat, and safety glasses
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers for damage
- Open shipping containers in adequately ventilated areas to assure worker safety
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the Cooler Receipt Form
- Compare samples received against those listed on the COC
- Verify that sample holding times have not been exceeded
- Examine all shipping records for accuracy and completeness
- Determine sample pH (if required for the scheduled analysis) (except VOA and TOX samples) and record on the Cooler Receipt Form (CRF)
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the coolers and samples on the cooler receipt form and notify the PM who in turn notifies the client

- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
- Place the samples in proper laboratory storage.

A Cooler Receipt Form (CRF) or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client shall be notified by the PM or designee of all CRFs generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. CRFs must be resolved prior to sample preparation and analysis. The completed CRF shall be stored in the project file. An example CRF is shown in Figure 24-4. The report narrative will include an explanation of sample receiving related anomalies.

24.2.2 Exceptions or Discrepancies

TestAmerica reserves the right to reject samples for any of the following reasons:

- No custody seals as required by project
- No chain of custody documentation provided
- Preservation inappropriate for analysis requested
- Sample container inappropriate for analysis requested
- Sample received out of holding time for analysis requested
- Incomplete sample information provided
- Discrepancies between COC and sample labels
- Samples have high levels of polychlorinated dibenzo-p-dioxins/ dibenzo furans (PCDD/PCDFs)
- Samples have a high level gross alpha or beta radiation
- Samples are from a site known to contain chemical warfare agents (CWAs) and the samples have not been screened for them.
- Samples containing high levels of PCBs, Cyanides, Sulfides, and Hydrofluoric Acid.
- Tissue samples that may contain viruses harmful to humans.
- Samples with percent levels of Target analytes must be discussed in advance before receipt.

These or any other project exceptions or discrepancies are discussed with the client and agreed upon action taken.

24.2.3 Sample Log-In

Sample log-in activities at TestAmerica North Canton are fully documented in SOP NC-SC-0005, Sample Receiving and Sample Control. The following is a general description of the log-in process:

- Client Name, Project Name, Address, Phone, Fax, Report to information, invoice to information. (Most of this information is "default information" that is stored in the LIMS.)
- Date and time sampled
- Date and time received
- Job and/or project description, sample description
- Sample matrix, special sample remarks
- Reporting requirements, i.e., QC level, report format, invoicing format
- Turn-around-time requirements
- Parameters (methods and reporting limits or MDLs are default information for a given parameter)

24.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy outlined in SOP NC-SC-0005, Sample Receiving and Sample Control, that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- A COC filled out completely
- Samples must be properly labeled
- Proper sample containers with adequate volume for the analysis and necessary QC
- Samples must be preserved according to the requirements of the requested analytical method
- Sample holding times must be adhered to
- All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time
- The Project Manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

24.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. Metals samples may be unrefrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

The primary considerations for sample storage are:

- Maintenance at the method prescribed temperature, if required
- Maintenance of sample integrity through adequate protection from contamination from outside sources or from cross-contamination of samples. Low-level and high-level samples,

when known, must be stored separately. Samples and standards must be stored in separate refrigerators or freezers. Storage areas for volatile organic test requests must be monitored weekly by the analysis of a holding (refrigerator) blank (an aliquot of contaminant-free water stored in a VOA vial)

- Security of samples within the laboratory.

The requirements listed in Tables 23-1 through 23-3 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of sample control personnel. TestAmerica will assign individuals the responsibility of notifying the Group/Team Leaders or their designees if there are any samples which must be analyzed immediately because of holding time requirements.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

24.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

All samples per SOP are treated as hazardous. If any extra or known hazards are present in the sample, the sample is flagged and precautions / instructions are put in the comments. Hazardous samples are segregated out, and go into the waste stream profile for the nature of the hazard. All soils--foreign and domestic--go to a USDA approved incinerator.

24.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The Chain-of-Custody form is signed by the Sample Control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

24.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP NC-SC-0005, Sample Receiving and Sample Control, and the Facility Addendum to the Corporate

Safety Manual.) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), and names of individuals who conducted the arrangements and physically completed the task. Sample labels are destroyed through the disposal method, e.g., samples are incinerated. A Waste Manifest is completed.

Figure 24-1. Example: Chain of Custody (COC)

[illegible]

Figure 24-2.

Example: Custody Seals

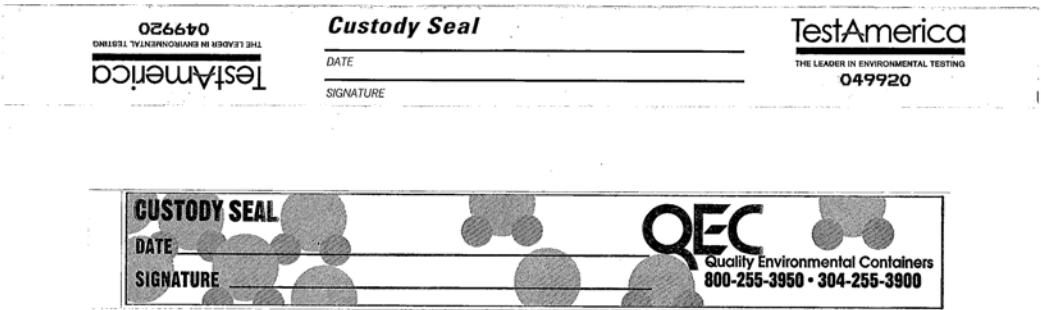


Figure 24-3.

Example: Internal Chain of Custody (COC)

**TestAmerica Laboratories, Inc.
Sample Control Record**

Client:

Lot Number:

Case Number/SDG:

Storage Location:

Laboratory Sample ID	Transferred By	Date	Entered	Removed	Reason	Date Returned

Figure 24-4.

Example: Cooler Receipt Form (Page 1)

TestAmerica Cooler Receipt Form/Narrative North Canton Facility		Lot Number: _____	
Client _____		Project _____ Quote # _____	
Cooler Received on _____		Opened on _____ By _____	
FedEx <input type="checkbox"/> Client Drop Off <input type="checkbox"/> UPS <input type="checkbox"/>		DHL <input type="checkbox"/> FAS <input type="checkbox"/> TestAmerica Courier <input type="checkbox"/>	
Stetson <input type="checkbox"/> US Cargo <input type="checkbox"/>		Other _____ (Signature)	
TestAmerica Cooler # _____		Foam Box <input type="checkbox"/> Client Cooler <input type="checkbox"/> Other _____	
1. Were custody seals on the outside of the cooler? Yes <input type="checkbox"/> No <input type="checkbox"/>		Intact? Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/>	
If YES, Quantity _____			
Were custody seals on the outside of cooler signed and dated?		Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/>	
Were custody seals on the bottles?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
If YES, are there any exceptions _____			
2. Shipper's packing slip attached to this form?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
3. Did custody papers accompany the sample(s)? Yes <input type="checkbox"/> No <input type="checkbox"/>		Relinquished by client? Yes <input type="checkbox"/> No <input type="checkbox"/>	
4. Did you sign the custody papers in the appropriate place?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
5. Packing material used: Bubble Wrap <input type="checkbox"/> Foam <input type="checkbox"/> None <input type="checkbox"/>		Other _____	
6. Cooler temperature upon receipt _____ °C (see back of form for multiple coolers/temps)			
METHOD: IR <input type="checkbox"/> Other <input type="checkbox"/>			
COOLANT: Wet Ice <input type="checkbox"/> Blue Ice <input type="checkbox"/> Dry Ice <input type="checkbox"/> Water <input type="checkbox"/> None <input type="checkbox"/>			
7. Did all bottles arrive in good condition (Unbroken)?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
8. Could all bottle labels and/or tags be reconciled with the COC?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
9. Were samples at the correct pH upon receipt?		Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/>	
10. Were correct bottles used for the tests indicated?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
11. Were air bubbles >6 mm in any VOA vials?		Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/>	
12. Sufficient quantity received to perform indicated analyses?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
13. Was a Trip Blank present in the cooler? Yes <input type="checkbox"/> No <input type="checkbox"/>		Were VOAs on the COC? Yes <input type="checkbox"/> No <input type="checkbox"/>	
Contacted PM _____ Date _____ by _____ via Voice Mail <input type="checkbox"/> Verbal <input type="checkbox"/> Other <input type="checkbox"/>			
Concerning _____			
14. CHAIN OF CUSTODY			
The following discrepancies occurred: _____ _____ _____			
15. SAMPLE CONDITION			
Sample(s) _____ were received after the recommended holding time had expired.			
Sample(s) _____ were received in a broken container.			
16. SAMPLE PRESERVATION			
Sample(s) _____ were further preserved in sample receiving to meet recommended pH level(s). Nitric Acid Lot #071707-HNO ₃ - Sulfuric Acid Lot # 092006-H ₂ SO ₄ ; Sodium Hydroxide Lot # 122805 -NaOH; Hydrochloric Acid Lot # 092006-HCl; Sodium Hydroxide and Zinc Acetate Lot # 050205-CH ₃ COO ₂ ZN/NaOH			
What time was preservative added to sample(s)? _____			
Sample(s) _____ were received with bubble > 6 mm in diameter (Notify PM)			
Client ID	pH	Date	Initials

Example: Cooler Receipt Form (Page 2)

[illegible]

SECTION 25.0

ASSURING THE QUALITY OF TEST RESULTS (NELAC 5.5.9)

25.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 21, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

25.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps may include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

25.3 NEGATIVE CONTROLS

25.3.1 Method Blanks are used to assess preparation and analysis for possible contamination during the preparation and processing steps.

25.3.1.1 The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.

25.3.1.2 The method blank goes through all of the steps of the process, including as necessary, filtration, clean-ups, etc.

25.3.1.3 The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is one for each batch of samples--not to exceed 20 environmental samples.

25.3.1.4 Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis. Generally, corrective action is taken if the concentration of a target analyte in the blank is at or above the reporting limit as established by the method or regulation. Refer to Policy QA-003, TestAmerica North Canton Quality Control Program.

- The source of contamination is investigated
- Measures are taken to minimize or eliminate the source of the contamination
- Affected samples are reprocessed or the results are qualified on the final report.

25.3.2 **Calibration Blanks** are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

25.3.3 **Instrument Blanks** are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

25.3.4 **Trip Blanks** are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. Trip Blanks are also sometimes referred to as Travel Blanks.

25.3.5 **Field Blanks** are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

25.3.6 **Equipment Blanks** are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

25.3.7 **Holding Blanks**, also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory. Refer to SOP NC-QA-0020, Laboratory Holding Blanks, for additional information on holding blank criteria.

25.3.8 **Field blanks**, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. When known, blanks should not be selected for matrix QC, as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB".

25.4 **POSITIVE CONTROLS**

Control samples, e.g., QC indicators, are analyzed with each batch of samples to evaluate data based upon:

- 1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps
- 2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

25.4.1 Method Performance Control - Laboratory Control Sample (LCS)

- 25.4.1.1** The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.
- 25.4.1.2** The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.
- 25.4.1.3** Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).
- 25.4.1.4** As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
- 25.4.1.5** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally one for each batch of samples; not to exceed 20 environmental samples.
- 25.4.1.6** If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable, e.g., no spike of pH. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in

Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

25.4.1.6.1 For methods that have 1-10 target analytes, spike all components.

25.4.1.6.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

25.4.1.6.3 For methods with more than 20 target analytes, spike at least 16 components.

25.4.1.6.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

25.4.1.6.5 Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.

25.4.1.7 **Accuracy Calculation:** Percent Recovery (%R) Calculation (applies to LCS, CCV, Surrogates, and Matrix Spikes.

$$\%R = \frac{AV}{TV} \times 100$$

Where: AV = Analyzed Value
TV = True Value

25.5 **SAMPLE MATRIX CONTROLS**

25.5.1 **Matrix Spikes (MS)**

25.5.1.1 The Matrix spike is used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used.

25.5.1.2 An MS is essentially a sample fortified with a known amount of the test analyte(s). At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.

25.5.1.3 If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number of the listed

components (see LCS analytes in Section 25.4.1.6 above) may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit-specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- 25.5.1.4** The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in Section 25.4.1.7, except that:

$$AV = Sp - Sa$$

Where: Sp = Spike result
Sa = Sample result

25.5.2 Surrogate Spikes

- 25.5.2.1** Surrogate Spikes are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.

- 25.5.2.2** Surrogate compounds are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method (also refer to Section 25.5). Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.

25.5.3 Duplicates

- 25.5.3.1** For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

- 25.5.3.2 Precision Calculation** (Relative Percent Difference - RPD)

$$RPD = \frac{|S - D|}{\frac{(S + D)}{2}} \times 100$$

Where: S=Sample Concentration
D=Duplicate Concentration

25.5.4 Internal Standards

- 25.5.4.1** In most organic analyses, internal standards are spiked into all environmental and quality control samples (including the initial calibration standards). An internal standard is also used with some metals analyses. It is added to sample extracts after the extraction (post-prep). The acceptance criteria in most methods are 50% to 200% of the responses in the mid-point of the corresponding calibration curve. Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria.
- 25.5.4.2** When the internal standard recoveries fall outside these limits, if there are not obvious chromatographic interferences, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets internal standard recovery criteria, the second run is reported (or both are reported if requested by the client).

25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

25.6.1 Each individual analyte in the LCS, MS, or Surrogate Spike are evaluated against the control limits as published in the test method. Where there are no established acceptance criteria, the laboratory calculates control limits with the use of control charts or, in some cases, utilizes client project specific or regulatory mandated control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data, e.g., unusual matrices not analyzed often, interim limits are established using available data or by analogy to similar methods or matrices.

25.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating (e.g. EPA SW846 8000 series methods). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

25.6.2.1 The lab should consider the effects of the spiking concentration control limits, and to avoid censoring of data. The acceptance criteria for recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling data to generate control limits.

25.6.2.2 Not only should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should all be generated using the same set of extraction, cleanup and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.

25.6.2.3 The laboratory should try and avoid discarding data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria, will lead to unrealistically narrow criteria. For a 99% confidence interval, one out of every 100 observations likely will still fall outside

the limits. For methods with long analyte lists this may mean occasional failures every batch or two. While professional judgment is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. However, data points shall be discarded if they were the result of human or mechanical error or sample concentration exceeded spike level by $> 4x$.

25.6.3 Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

25.6.3.1 Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

25.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method.

25.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable).

25.6.3.4 The maximum acceptable recovery limit will be 200%.

25.6.3.5 The maximum acceptable RPD limit will be 30% for organic methods and 20% for inorganic methods. The minimum RPD limit is 10%.

25.6.3.6 If either the high or low end of the control limit changes by $\leq 10\%$ from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

25.6.4 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Refer to NC-QA-0018, Statistical Evaluation of Data and Development of Control Charts, for details.

25.6.4.1 One example: The QA Department generates a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses. Unless otherwise noted, limits within these tables are laboratory generated. Once reviewed and approved, the limits are entered into LIMS and are effective immediately. The Quality Assurance department maintains an archive of all limits used within the laboratory.

25.6.5 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

25.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

25.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

25.6.5.3 Or, for NELAC and Department Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):

- <11 analytes 0 marginal exceedances are allowed.
- 11 – 30 Analytes 1 marginal exceedance is allowed
- 31-50 Analytes 2 marginal exceedances are allowed
- 51-70 Analytes 3 marginal exceedances are allowed
- 71-90 Analytes 4 marginal exceedances are allowed
- > 90 Analytes 5 marginal exceedances are allowed

25.6.5.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).

25.6.5.3.2 Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

25.6.5.3.3 Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

25.6.6 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in Appendix 4 and in Section 13.

25.6.7 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

25.7 METHOD DETECTION LIMITS (MDLs)

MDLs, calculated as described in Section 20.7, are updated or verified annually, or more often if required by the method.

25.8 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

25.8.1 The laboratory has written procedures to assure the accuracy of the test method including calibration (see Section 21), use of certified reference materials (see Section 22), and use of PT samples (see Section 16).

25.8.2 A discussion regarding MDLs, Limit of Detection (LOD), and Limit of Quantitation (LOQ) can be found in Section 20.

25.8.3 Use of formulae to reduce data is discussed in the method Standard Operating Procedures and in Section 21.

25.8.4 Selection of appropriate reagents and standards is included in Sections 9 and 22.

25.8.5 A discussion on selectivity of the test is included in Section 5.

25.8.6 Constant and consistent test conditions are discussed in Section 19.

25.8.7 The laboratory sample acceptance policy is included in Section 24.

SECTION 26.0

REPORTING RESULTS (NELAC 5.5.10)

26.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is a conflict between the client requested formats and accreditation requirements or data usability information, accreditation requirements and data usability information will take precedence over client requests. A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 20.

26.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed, reviewed, and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:

26.2.1 A report title with a "Sample Result" header.

26.2.2 Each report page printed includes the laboratory name, address, and telephone number.

26.2.3 A unique identification of the report, e.g. Work Order number, and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented at the bottom of each page. The report is sequentially paginated. The final page of the report is labeled as "End of Report".

26.2.4 A copy of the Chain-of-Custody (COC).

- Any COCs involved with subcontracting are included.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g. Sampling information).

26.2.5 The name and address of client and a project name/number, if applicable.

26.2.6 Client project manager or other contact

26.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

26.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

26.2.9 Date reported or date of revision, if applicable

26.2.10 Method of analysis including method code (EPA, Standard Methods, etc)

26.2.11 Reporting limit

26.2.12 Method detection limits (if requested)

26.2.13 Definition of Data qualifiers and reporting acronyms, e.g., ND

26.2.14 Sample results

26.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits

26.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (refer to Section 26.2.4 – Item 3, regarding additional addenda).

26.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

26.2.18 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

26.2.19 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

26.2.20 The laboratory includes a cover page.

26.2.21 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

26.2.22 When Soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

26.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

26.2.24 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report, e.g., partial report, or how your lab identifies it. A complete report will follow once all of the work has been completed.

26.2.25 Any out-of-network subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All in-network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

26.3 REPORTING LEVEL OR REPORT TYPE

TestAmerica North Canton offers two levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Standard report – all features in Section 26.2
- Expanded deliverable – standard report

Presented on CLP-like forms and relevant calibration information. All supporting raw data is supplied.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Procedures used to ensure client confidentiality are outlined in Section 26.7.

26.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica’s services. TestAmerica North Canton offers a variety of EDD formats.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

26.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report. Refer to Appendix 8 for a list of the laboratory's standard footnotes and qualifiers.

26.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

26.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

26.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

26.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

26.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If TestAmerica North Canton is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

26.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore,

information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

26.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

"Confidentiality Notice: The information contained in this message is intended only for the use of the addressee, and may be confidential and/or privileged. If the reader of this message is not the intended recipient, or the employee or agent responsible to deliver it to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify the sender immediately."

26.7 **FORMAT OF REPORTS**

The format of reports are designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

26.8 **AMENDMENTS TO TEST REPORTS**

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 13).

If, after issuance of a report, TestAmerica North Canton observes any mistake that affects the results reported or the QC interpretation of those results, the client will be notified. After issuance of the report, the laboratory report remains unchanged. Any material amendments to a report after issue made only in the form of a further document, or data transfer must include the statement "Supplement to Test Report" or otherwise identified.

When the report is re-issued, a notation is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the re-issue.

26.9 **POLICIES ON CLIENT REQUESTS FOR AMENDMENTS**

26.9.1 **Sample Reanalysis Policy**

Because there is a certain level of uncertainty with any analytical measurement a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are

also variables that may be present (e.g. sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific arrangements for reanalysis protocols can be established.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples $\leq 5\times$ the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Group Leader, QA Manager, or Laboratory Director/Manager if unsure.

26.9.2 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results, including data qualifiers, or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested, e.g., COC lists 8315 but client wanted 8310. A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely no possible impact on the interpretation of the analytical results and there is no possibility of the change being interpreted as misrepresentation by anyone inside or outside of our company.

26.9.3 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. TestAmerica Ethics Policy CA-L-P-001

**TESTAMERICA
ETHICS POLICY No. CA-L-P-001**

Refer to CA-L-P-001 for complete policy.

**TestAmerica
EMPLOYEE ETHICS STATEMENT**

I understand that TestAmerica is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

- With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:*
- I will not intentionally report data values that are inconsistent with the actual values observed or measured.*
- I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations.*
- I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's.*
- I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.*
- I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers. I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions.*
- I shall not compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica laboratory, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.*
- I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Quality Assurance Manager. The Quality Assurance Manager will initial and date the information and return a copy to me. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.*

- *I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices, or if I am in doubt or uncertain as to whether or not such laboratory practices are proper, I will not comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Lab Director, all supervisors and managers with direct line reporting relationship between me and the Lab Director, and the local Quality Assurance representative, excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.*
- *I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or retained by TestAmerica for subsequent internal use;*
- *I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.*
- *I shall not accept gifts of a value that would adversely influence judgment.*
- *I shall avoid conflicts of interest and report any potential conflicts to the management (e.g. employment or consulting with competitors, clients, or vendors).*
- *I shall not participate in unfair competition practices (e.g. slandering competitors, collusion with other labs to restrict others from bidding on projects).*
- *I shall not misrepresent certifications and status of certifications to clients or regulators.*
- *I shall not intentionally discharge wastes illegally down the drain or onto the ground.*
- *I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.*

As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination of my employment. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE _____

Date _____

Supervisor/Trainer: _____

Date _____

Work Instruction No. CA-WI-005

TestAmerica
CONFIDENTIALITY AND PROPRIETARY INFORMATION AGREEMENT

TestAmerica and their predecessors, in their businesses, have developed and use commercially valuable technical and non-technical information and to guard the legitimate interests of TestAmerica and its clients, it is necessary to protect certain information as confidential and proprietary.

I, _____, understand and acknowledge that during the term of my employment by TestAmerica, I will be privy to and entrusted with certain confidential information and trade secrets of TestAmerica and its clients.

Confidential information and trade secrets include, but are not limited to: customer and client lists; price lists; marketing and sales strategies and procedures; operational and equipment techniques; standard operating procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; the trade secrets of clients; client's data; vendor or supplier pricing; employee lists and personal information, and any other records, data, files, drawings, inventions, discoveries, applications, or processes which are not in the public domain.

I agree as follows:

1. I will not in any way, during the term of my employment, or at any time thereafter, except as authorized in writing by the Legal Department of TestAmerica or the client where client data is involved, disclose to others, use for my own benefit, remove from TestAmerica's premises (except to the extent off-site work is approved by my supervisor), copy or make notes of any confidential information and/or trade secrets of TestAmerica or its clients, excepting only that information which may be public knowledge. Technical and business information of any previous employer or other third party which I may disclose to TestAmerica shall be limited to that which was acquired legitimately and disclosed to me without restriction as to secrecy.
2. I agree that all inventions (whether or not patentable) conceived or made by me during the period of my employment by TestAmerica shall belong to TestAmerica, provided such inventions grow out of my work for TestAmerica and are related to the business of TestAmerica. I agree to disclose and assign such inventions to TestAmerica. In California, this provision shall not apply to any invention which qualifies fully under Section 2870 of the California Labor Code.
3. On termination of my employment from TestAmerica, I will deliver to TestAmerica all documents, records, notes, data, memoranda, files, manuals, equipment and things of any nature which relate in any way to confidential information and/or trade secrets of TestAmerica or its clients and which are in my possession or under my control.
4. I agree that during the period of my employment and for one (1) year from and after the termination (for any reason) of my employment with TestAmerica, I shall not directly or indirectly (without first obtaining the written permission of TestAmerica), recruit for employment, or induce to terminate his or her employment with TestAmerica, any person who is an active employee of TestAmerica on the last day of my employment with TestAmerica.
5. I acknowledge that if I were to breach any provision of this Confidentiality Agreement, money damages will be inadequate, and I hereby agree that TestAmerica shall be entitled, where appropriate, to specific performance and/or injunctive relief (i.e. to require me to comply with this Agreement). I further acknowledge that the willingness of TestAmerica to hire me or to continue my employment constitutes full and adequate consideration for the agreements, and obligations to which I have agreed as set forth in this document.

I have executed this Agreement, intending to be legally bound.

Printed Name

Signature

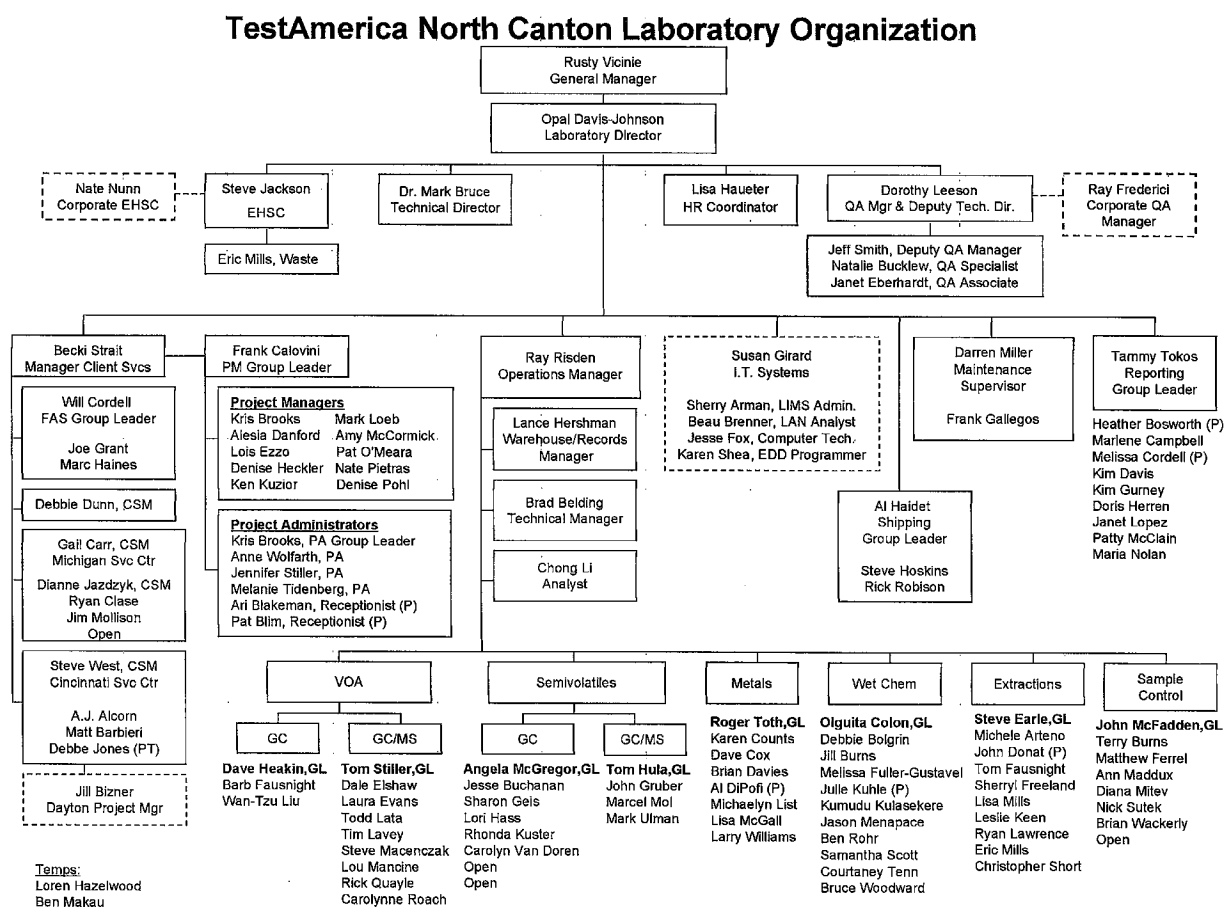
Date

Work Instruction No. CA-WI-006

Appendix 2.

Example Laboratory Organization Chart

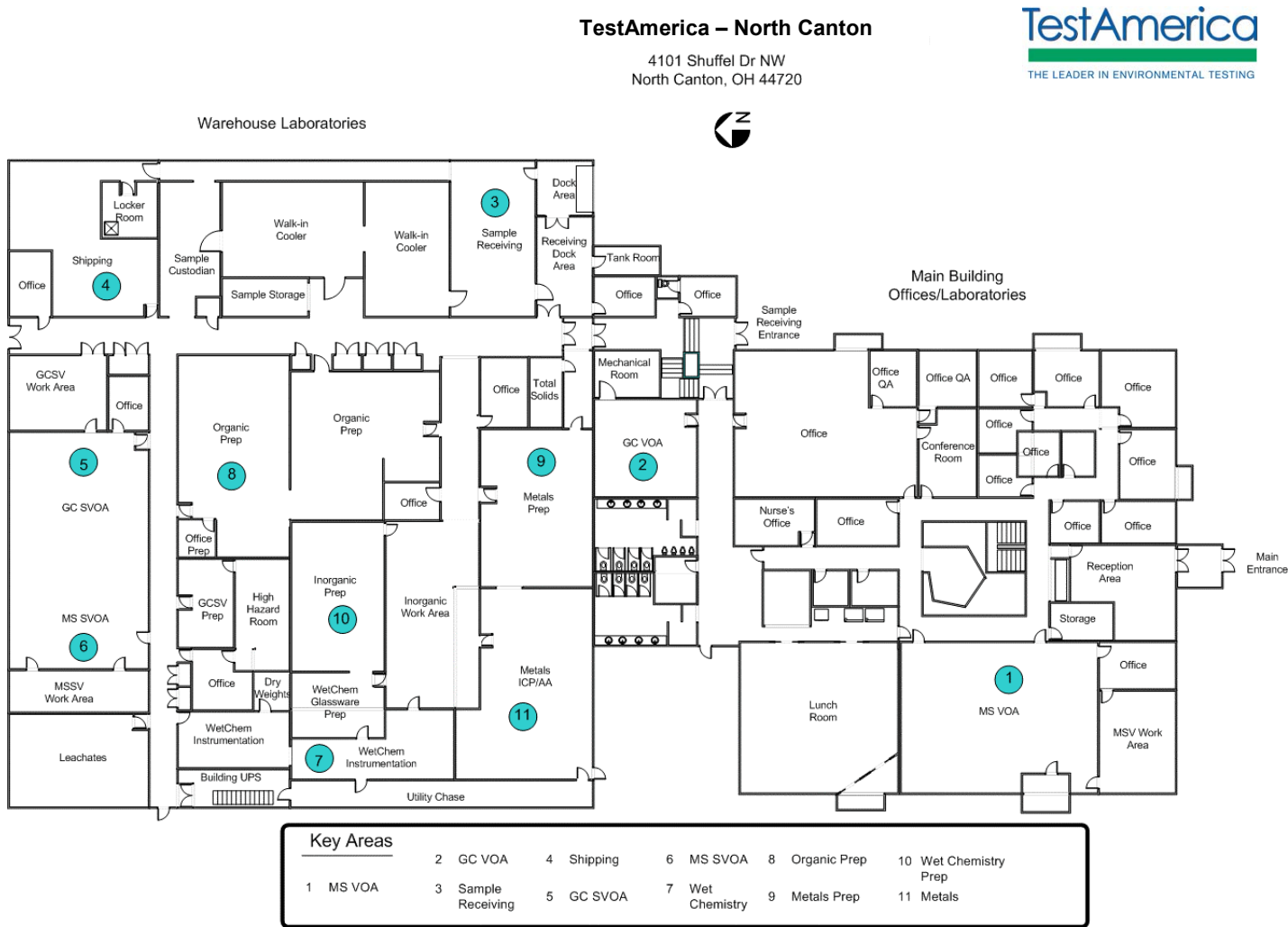
(The most current chart can be obtained from the QA Manager or Lab Director/Manager)



11/06/07

Appendix 3.

Laboratory Floor Plan



Appendix 4.

Laboratory Method Listing

Wet Chemistry Methods ¹

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Acidity	Water		305. ² SM 2310 B	--	--
Alkalinity, Bicarbonate, Carbonate	Water		305. ² SM 2320 B		
	Solid		EPA 310.1 ² (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water		--	EPA 7063	--
Ash Content	Solid		--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water		EPA 405.1 SM 5210 B	--	--
Bromide	Water		EPA 300.0A	EPA 9056A	--
	Waste		EPA 300.0A	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A	--
Cation-Exchange Capacity	Solid		--	EPA 9081	--
Chemical Oxygen Demand	Water		EPA 410.4 SM 5220D	--	--
	Waste		EPA 410.4	--	--
Chloride	Water		EPA 300.0A EPA 325.2 ²	EPA 9056A EPA 9251	EPA 325.2 ²
	Waste		EPA 300.0A	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A EPA 9251(M)	--
Chromium, Hexavalent	Water		EPA 3500-Cr-D	EPA 7196A	--
	Waste		EPA 3500-Cr-D	EPA 7196A	--
	Solid		--	EPA 3060A EPA 7196A	--

¹ Any matrix not listed is not applicable for the associated method

² Removed from 40CFR

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Specific Conductance	Water		EPA 120.1 SM 2510B	EPA 9050A	--
	Waste		EPA 120.1	EPA 9050A	--
	Solid		--	EPA 9050A	--
Chlorine, Residual	Water		EPA 330.5 ² SM 3500 CL-G	--	--
Cyanide (Amenable)	Water		EPA 335.1 ² SM 4500 CN-G	EPA 9012A	--
	Solid		--	EPA 9012A	--
Cyanide (Total)	Water		SM 4500-CN E EPA 335.4	EPA 9012A	---
	Waste		--	EPA 9012A	--
	Solid		--	EPA 9012A	--
Cyanide (Weak and Dissociable) (Free)	Water		SM 4500-CN I	--	--
Dissolved Oxygen	Water		360.1 ² SM 4500 O-G	--	--
Flash Point	Waste		--	EPA 1010	ASTM D93-9
	Solid		--	EPA 1010	ASTM D93-9
Fluoride	Water		EPA 300.0A EPA 340.2 ²	EPA 9056A	SM 4500 F-C, ISE
	Waste		EPA 340.2 (M) ² EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 340.2 (M) ² EPA 300.0A (M)	EPA 9056A	--
Iron, Ferrous & Ferric	Water		SM 3500 FE D	--	--
Hardness	Water		EPA 130.2 ²	--	SM 2340B
Moisture	Solid		---	EPA 160.3 (M) ASTM D2216-90	---
Nitrogen, Ammonia	Water		EPA 350.1	--	EPA 350.2 ²
	Waste		EPA 350.1	--	EPA 350.2 ²
	Solid		EPA 350.1	--	EPA 350.2 ²
	Water		SM 4500 NH ₃ -B (Distillation)	--	--
	Water		SM 4500 NH ₃ -E (Titration)	--	--
	Water		SM 4500 NH ₃ -F (ISE)	--	--

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Nitrite (NO ₂)	Water		EPA 300.0A	EPA 9056A	--
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A	--
Nitrate (NO ₃)	Water		EPA 300.0A	EPA 9056A	SM 4500 NO ₃ -E
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M)	--	--
Nitrate plus Nitrite NO ₂ /NO ₃	Water		EPA 353.2	--	--
	Waste		EPA 353.2	--	--
Total Kjeldahl Nitrogen (TKN)	Water		EPA 351.3	--	SM 4500 NO ₃
	Waste		EPA 351.3	--	--
	Solid		EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water		EPA 1664A	EPA 9071B	--
	Waste		EPA 1664A	EPA 9071B	--
	Solid		--	EPA 9071B	--
Ortho-phosphate o-PO ₄	Water		EPA 300.0A EPA 365.1	EPA 9056A	SM 4500 P-E
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M) EPA 365.1	EPA 9056A	--
pH	Water		EPA 150.1 ²	EPA 9040B	EPA 9041
	Waste		SM 4500 H-B	EPA 9045C	--
	Solid		---	EPA 9045C	--
Paint Filter	Water		--	EPA 9095A	--
Phenolics	Water		EPA 420.1	--	--
	Waste		--	EPA 9065	--
	Solid		--	EPA 9065	--
Phosphorus (Total)	Water		EPA 365.1	--	SM 4500 P-E
	Waste		EPA 365.1	--	--
	Solid		EPA 365.1	--	--
Sulfate (SO ₄)	Water		EPA 300.0A EPA 375.4 ²	EPA 9056A EPA 9038	--
	Waste		EPA 300.0A (M) EPA 375.4 ²	EPA 9056A EPA 9038	--
	Solid		EPA 300.0A (M)	EPA 9056A EPA 9038 (M)	--

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA	Other
Sulfide	Water		EPA 376.1 ²	EPA 9030A SM 4500	9030B/9034
Total Organic Carbon (TOC)	Water		EPA 415.1 ²	EPA 9060	SM 5310 D
	Waste		--	EPA 9060	--
	Solid		EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water		--	EPA 9020B EPA 9023(EOX)	EPA 450.1
	Waste		--	--	--
	Solid		--	EPA 9020B	--
Total Petroleum Hydrocarbons	Water		EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste		EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid		--	EPA 9071B	--
Total Solids	Water		EPA 160.3	--	--
	Waste		EPA 160.3	--	--
	Solid		EPA 160.3 (M)	--	--
Total Dissolved Solids	Water		EPA 160.1	--	2540E
Total Suspended Solids	Water		EPA 160.2	---	2540E
Volatile and Volatile Suspended Solids	Water		EPA 160.4	--	--
Settleable Solids	Water		EPA 160.5	--	--
Turbidity	Water		EPA 180.1	--	--

Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water		EPA 245.1	EPA 7470A	--
	TCLP Leachate		--	EPA 7470A	--
	Waste		--	EPA 7471A	--
	Solid		EPA 254.5	EPA 7471A	--

Methods for Mercury by Cold Vapor Atomic Fluorescence

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water		EPA 245.7	--	EPA 1631E

Methods for Metals by ICP and ICPMS

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Aluminum	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Antimony	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Arsenic	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Barium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Beryllium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Boron	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---
Calcium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Cadmium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Cobalt	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Chromium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Copper	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Iron	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Lead	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Magnesium	Water		EPA 200.7	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Manganese	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Molybdenum	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Nickel	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Potassium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Selenium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Silver	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Sodium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Tin	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Thallium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Titanium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Vanadium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		---	EPA 6010B EPA 6020	---
Zinc	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Metals Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water		---	EPA 1311	---
	Waste		---	EPA 1311	---
	Solid		---	EPA 1311	---
ICP Metals	Water		EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
ICPMS Metals	Water		EPA 200.8	EPA 3010A	---
	TCLP		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
CVAA Mercury	Water		EPA 245.1	EPA 7470A	---
	TCLP Leachate		---	EPA 7470A	---
	Waste		---	EPA 7471A	---
	Solid		---	EPA 7471A	---
CVAFS Mercury Low Level	Water		EPA 245.7	---	EPA 1631E

Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water		EPA 624	EPA 5030B	---
	Waste		---	EPA 5030B EPA 5035	---
	Solid		---	EPA 5035 EPA 5035A	---
Halogenated Volatiles by GC	Water		EPA 601	EPA 5030B	---
	Waste		---	EPA 5030B EPA 5035	---
	Solid		---	EPA 5035 EPA 5035A	---
Aromatic Volatiles by GC	Water		EPA 602	EPA 5030B	---
	Waste		---	EPA 5030B EPA 5035	---
	Solid		---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water		EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate		---	EPA 3510C EPA 3520C	---
	Waste		---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid		---	EPA 3550B EPA 3540C EPA 3541	---
Pesticides/PCBs by GC	Water		EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate		---	EPA 3510C EPA 3520C	---
	Waste		---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid		---	EPA 3550B EPA 3540C EPA 3541	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Herbicides by GC	Water		EPA 615	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water		---	EPA 5030B	WI GRO
	Waste		---	EPA 5030B EPA 5035	WI GRO
	Solid		---	EPA 5035 EPA 5035	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water		---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate		---	EPA 3510C EPA 3520C	---
	Waste		---	EPA 3550B EPA 3580A	WI DRO
	Solid		---	EPA 3550B	WI DRO

Organic Methods of Analysis

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water		EPA 624	EPA 8260B	---
	Waste		---	EPA 8260B	---
	Solid		---	EPA 8260B	---
Halogenated Volatiles by GC	Water		EPA 601	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Aromatic Volatiles by GC	Water		EPA 602	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Semivolatiles by GC/MS	Water		EPA 625	EPA 8270C	
	Waste		---	EPA 8270C	---
	Solid		---	EPA 8270C	---
Pesticides/PCBs by GC	Water		EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate		---	Pesticides 8081A PCBs 8082	---
	Waste		---	Pesticides 8081A PCBs 8082	---
	Solid		---	Pesticides 8081A PCBs 8082	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Phenoxyacid Herbicides by GC	Water		---	EPA 8151A	---
	TCLP Leachate		---	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Gasoline Range Organics by GC	Water		---	EPA 8015B (M)	WI GRO
	Waste		---	EPA 8015B (M)	---
	Solid		---	EPA 8015B (M)	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID Dissolved Gases RSK-175	Water		---	EPA 8015B (M)	WI DRO
	Waste		---	EPA 8015B (M)	---
	Water		---	---	SOP
Formaldehyde Carbonyl Compounds	Water		---	EPA 8315	---

Appendix 5.

Laboratory Reporting Limits

**General Chemistry
Reporting Limits (RL), Method Reference**

Test Method	Analyte	RL	Units
305.1/SM2310B	Acidity	5	mg/L
310.1/SM2320B	Alkalinity, Total	5	mg/L
7063	Arsenic Speciation	2	ug/L
300.0 9056A	Bromide Chloride Nitrate Fluoride Sulfate Ortho-Phosphate Nitrite	0.5 1 0.1 1 1 0.5 0.1	mg/L mg/L mg/L mg/L mg/L mg/L mg/L
405.1/SM5210B	Biochemical Oxygen Demand	2	mg/L
410.4/SM5220D	Chemical Oxygen Demand (COD)	10	mg/L
325.2/9251	Chloride, Automated	1	mg/L
330.5 SM4500CL-G	Chlorine, Total Residue	0.2	mg/L
9012A/335.2 335.4 SM4500CN-E	Cyanide, Total	0.01	mg/L
340.2 SM4500F-C, ISE	Fluoride	0.1	mg/L
130.2/SM2340B	Hardness, as CaCO ₃	5	mg/L
1664A	n-Hexane Extractable Material n-Hexane Extractable Material, SGT	5 10	mg/L mg/L
7196A	Hexavalent Chromium	0.02	mg/L
3500 Cr D	Hexavalent Chromium	0.02	mg/L
353.2 SM4500NO ₃ E	Nitrite Nitrate Nitrate/Nitrite	0.1 0.1 0.1	mg/L mg/L mg/L
350.2 SM4500NH ₃ -E,F	Nitrogen, as Ammonia	1	mg/L

General Chemistry
Reporting Limits (RL), Method Reference

Test Method	Analyte	RL	Units
9065	Phenols, Total	0.02	mg/L
9045B, C SM4500H-B	pH (solid)	--	No Units
4500	Phosphorus as Orthophosphate	0.1	mg/L
365.2 365.1	Ortho-Phosphate Phosphorus, as ortho-Phosphate ortho-Phosphate, Dissolved	0.1 0.1 0.1	mg/L mg/L mg/L
120.1 SM2510B	Specific Conductance	1	umhos/cm
9050A	Specific Conductance	1	umhos/cm
160.1 SM2540E	Solids (Residue), Dissolved Filterable Solids, Total Dissolved	10 10	mg/L mg/L
375.4	Sulfate	5	mg/L
9038	Sulfate	5	mg/L
376.1	Sulfide, Total	1	mg/L
9030A	Sulfide, Total	1	mg/L
9030A	Sulfide, Acid-Insoluble	1	mg/L
351.3	Total Kjeldahl Nitrogen	3	mg/L
415.1 SM5310D	Total Organic Carbon	1	mg/L
9060	Total Organic Carbon	1	mg/L
9020B	Total Organic Halogens	30	ug/L
180.1	Turbidity	0.5	NTU

Metals – ICP, CVAA 6000, 7000, 200 Series
Reporting Limits (RL), Method Reference^{1, 2}

	Analyte	RL	Units
Trace Water	Antimony	0.01	mg/L
	Arsenic	0.01	mg/L
	Cadmium	0.002	mg/L
	Chromium	0.005	mg/L
	Cobalt	0.007	mg/L
	Lead	0.003	mg/L
	Molybdenum	0.01	mg/L
	Selenium	0.005	mg/L
	Silver	0.005	mg/L
	Thallium	0.01	mg/L
	Vanadium	0.007	mg/L
Trace Solid	Antimony	1	mg/kg
	Arsenic	1	mg/kg
	Cadmium	0.2	mg/kg
	Chromium	0.5	mg/kg
	Cobalt	5	mg/kg
	Lead	0.3	mg/kg
	Molybdenum	1	mg/kg
	Selenium	0.5	mg/kg
	Silver	0.5	mg/kg
	Thallium	1	mg/kg
	Vanadium	5	mg/kg
Water	Aluminum	0.2	mg/L
	Antimony	0.06	mg/L
	Arsenic	0.3	mg/L
	Barium	0.2	mg/L
	Beryllium	0.005	mg/L
	Boron	0.2	mg/L
	Cadmium	0.005	mg/L
	Calcium	5	mg/L
	Chromium	0.01	mg/L
	Cobalt	0.05	mg/L
	Copper	0.025	mg/L
	Iron	0.1	mg/L
	Lead	0.1	mg/L
	Magnesium	5	mg/L
	Manganese	0.015	mg/L
	Molybdenum	0.04	mg/L
	Nickel	0.04	mg/L
	Potassium	5	mg/L
	Selenium	0.25	mg/L
	Silver	0.01	mg/L
	Sodium	5	mg/L
	Thallium	0.2	mg/L
	Tin	0.1	mg/L
	Titanium	0.05	mg/L
	Vanadium	0.05	Mg/L
	Zinc	0.02	mg/L

TABLE 8.2-10-2
Metals – ICP, CVAA 6000, 7000, 200 Series
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Solid	Aluminum	20	mg/kg
	Antimony	6	mg/kg
	Arsenic	30	mg/kg
	Barium	20	mg/kg
	Beryllium	0.5	mg/kg
	Boron	20	mg/kg
	Cadmium	0.5	mg/kg
	Calcium	500	mg/kg
	Chromium	1	mg/kg
	Cobalt	5	mg/kg
	Copper	2.5	mg/kg
	Iron	10	mg/kg
	Lead	10	mg/kg
	Magnesium	500	mg/kg
	Manganese	1.5	mg/kg
	Molybdenum	4	mg/kg
	Nickel	4	mg/kg
	Potassium	500	mg/kg
	Selenium	25	mg/kg
	Silver	1	mg/kg
	Sodium	500	mg/kg
	Thallium	200	mg/kg
	Tin	10	mg/kg
	Titanium	5	mg/kg
	Vanadium	5	mg/kg
	Zinc	2	mg/kg
Water	Mercury	0.0002	mg/L
Solid	Mercury	0.1	mg/kg

Metals – ICPMS, 6020
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aluminum	50	ug/L
	Antimony	2	ug/L
	Arsenic	5	ug/L
	Barium	1	ug/L
	Beryllium	1	ug/L
	Cadmium	1	ug/L
	Chromium	2	ug/L
	Cobalt	1	ug/L
	Copper	2	ug/L
	Iron	20	ug/L
	Lead	1	ug/L
	Manganese	1	ug/L
	Molybdenum*	10	ug/L
	Nickel	2	ug/L
	Selenium*	5	ug/L
	Silver	1	ug/L
	Thallium	1	ug/L
	Tin*	10	ug/L
	Vanadium*	5	ug/L
	Zinc	10	Ug/L

*Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

Solid	Antimony	0.2	mg/kg
	Arsenic	0.5	mg/kg
	Barium	0.1	mg/kg
	Beryllium	0.1	mg/kg
	Cadmium	0.1	mg/kg
	Chromium	0.2	mg/kg
	Cobalt	0.1	mg/kg
	Copper	0.2	mg/kg
	Lead	0.1	mg/kg
	Manganese	0.1	mg/kg
	Molybdenum*	0.2	mg/kg
	Nickel	0.1	mg/kg
	Selenium*	0.5	mg/kg
	Silver	0.1	mg/kg
	Thallium	0.1	mg/kg
	Tin*	1	mg/kg
	Vanadium*	0.5	mg/kg
	Zinc	1	mg/kg

*Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

Metals – ICPMS, 200.8
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aluminum	50	ug/L
	Antimony	2	ug/L
	Arsenic	5	ug/L
	Barium	1	ug/L
	Beryllium	1	ug/L
	Cadmium	1	ug/L
	Chromium	2	ug/L
	Cobalt	1	ug/L
	Copper	2	ug/L
	Iron	20	ug/L
	Lead	1	ug/L
	Manganese	1	ug/L
	Molybdenum	10	ug/L
	Nickel	2	ug/L
	Selenium	2	ug/L
	Silver	1	ug/L
	Thallium	1	ug/L
	Tin*	10	ug/L
	Vanadium	5	ug/L
	Zinc	10	ug/L

*Tin is not on the 200.8 method list

Metals – Low-Level Mercury, 1631E & 245.7
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
1631E Water	Mercury	0.5	ng/L
245.7 Water	Mercury	5	ng/L

MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Acenaphthene	10	ug/L
	Acenaphthylene	10	ug/L
	Anthracene	10	ug/L
	Benzo(a)anthracene	10	ug/L
	Benzo(b)fluoranthene	10	ug/L
	Benzo(k)fluoranthene	10	ug/L
	Benzo(ghi)perylene	10	ug/L
	Benzo(a)pyrene	10	ug/L
	Bis(2-chloroEthoxy)methane	10	ug/L
	bis(2-Chloroethyl) ether	10	ug/L
	bis(2-Ethylhexyl) phthalate	10	ug/L
	4-Bromophenyl phenyl ether	10	ug/L
	Butyl benzyl phthalate	10	ug/L
	Carbazole	10	ug/L
	4-Chloroaniline	10	ug/L
	4-Chloro-3-methylphenol	10	ug/L
	2-Chloronaphthalene	10	ug/L
	2-Chlorophenol	10	ug/L
	4-Chlorophenyl phenyl ether	10	ug/L
	Chrysene	10	ug/L
	Dibenz(a,h)anthracene	10	ug/L
	Dibenzofuran	10	ug/L
	Di-n-butyl phthalate	10	ug/L
	1,2-Dichlorobenzene	10	ug/L
	1,3-Dichlorobenzene	10	ug/L
	1,4-Dichlorobenzene	10	ug/L
	3,3'-Dichlorobenzidine	50	ug/L
	2,4-Dichlorophenol	10	ug/L
	Diethyl phthalate	10	ug/L
	2,4-Dimethylphenol	10	ug/L
	Dimethyl phthalate	10	ug/L
	4,6-Dinitro-2-methylphenol	50	ug/L
	2,4-Dinitrophenol	50	ug/L
	2,4-Dinitrotoluene	10	ug/L
	2,6-Dinitrotoluene	10	ug/L
	Di-n-octyl phthalate	10	ug/L
	Fluoranthene	10	ug/L
	Fluorene	10	ug/L
	Hexachlorobenzene	10	ug/L
	Hexachlorobutadiene	10	ug/L
	Hexachloro-cyclopentadiene	50	ug/L
	Hexachloroethane	10	ug/L
	Indeno(1,2,3-cd)pyrene	10	ug/L
	Isophorone	10	ug/L
	2-Methylnaphthalene	10	ug/L
	2-Methylphenol	10	ug/L
	4-Methylphenol	10	ug/L
	Naphthalene	10	ug/L
	2-Nitroaniline	50	ug/L

	Analyte	RL	Units
	3-Nitroaniline	50	ug/L
	4-Nitroaniline	50	ug/L
	Nitrobenzene	10	ug/L
	2-Nitrophenol	10	ug/L
	4-Nitrophenol	50	ug/L
	N-Nitrosodiphenylamine	10	ug/L
	N-Nitrosodi-n-propylamine	10	ug/L
	Pentachlorophenol	10	ug/L
	Phenanthrene	10	ug/L
	Phenol	10	ug/L
	2,2'-Oxybis(1-Chloropropane)	10	ug/L
	Pyrene	10	ug/L
	1,2,4-Trichlorobenzene	10	ug/L
	2,4,5-Trichlorophenol	10	ug/L
	2,4,6-Trichlorophenol	10	ug/L
Water Surrogates	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Solid	Acenaphthene	330	ug/kg
	Acenaphthylene	330	ug/kg
	Anthracene	330	ug/kg
	Benzo(a)anthracene	330	ug/kg
	Benzo(b)fluoranthene	330	ug/kg
	Benzo(k)fluoranthene	330	ug/kg
	Benzo(ghi)perylene	330	ug/kg
	Benzo(a)pyrene	330	ug/kg
	bis(2-Chloroethoxy)-methane	330	ug/kg
	bis(2-Chloroethyl) ether	330	ug/kg
	bis(2-Ethylhexyl) phthalate	330	ug/kg
	4-Bromophenyl phenyl ether	330	ug/kg
	Butyl benzyl phthalate	330	ug/kg
	Carbazole	330	ug/kg
	4-Chloroaniline	330	ug/kg
	4-Chloro-3-methylphenol	330	ug/kg
	2-Chloronaphthalene	330	ug/kg
	2-Chlorophenol	330	ug/kg
	4-Chlorophenyl phenyl ether	330	ug/kg
	Chrysene	330	ug/kg
	Dibenz(a,h)anthracene	330	ug/kg
	Dibenzofuran	330	ug/kg
	Di-n-butyl phthalate	330	ug/kg
	1,2-Dichlorobenzene	330	ug/kg
	1,3-Dichlorobenzene	330	ug/kg
	1,4-Dichlorobenzene	330	ug/kg
	3,3'-Dichlorobenzidine	1600	ug/kg
	2,4-Dichlorophenol	330	ug/kg
	Diethyl phthalate	330	ug/kg
	2,4-Dimethylphenol	330	ug/kg
	Dimethyl phthalate	330	ug/kg
	4,6-Dinitro-2-methylphenol	1600	ug/kg
	2,4-Dinitrophenol	1600	ug/kg
	2,4-Dinitrotoluene	330	ug/kg
	2,6-Dinitrotoluene	330	ug/kg
	Di-n-octyl phthalate	330	ug/kg
	Fluoranthene	330	ug/kg
	Fluorene	330	ug/kg
	Hexachlorobenzene	330	ug/kg
	Hexachlorobutadiene	330	ug/kg
	Hexachloro-cyclopentadiene	1600	ug/kg
	Hexachloroethane	330	ug/kg
	Indeno(1,2,3-cd)pyrene	330	ug/kg
	Isophorone	330	ug/kg
	2-Methylnaphthalene	330	ug/kg
	2-Methylphenol	330	ug/kg
	4-Methylphenol	330	ug/kg
	Naphthalene	330	ug/kg
	2-Nitroaniline	1600	ug/kg
	3-Nitroaniline	1600	ug/kg

	Analyte	RL	Units
	4-Nitroaniline	1600	ug/kg
	Nitrobenzene	330	ug/kg
	2-Nitrophenol	330	ug/kg
	4-Nitrophenol	1600	ug/kg
	N-Nitrosodiphenylamine	330	ug/kg
	N-Nitrosodi-n-propylamine	330	ug/kg
	Pentachlorophenol	330	ug/kg
	Phenanthrene	330	ug/kg
	Phenol	330	ug/kg
	2,2'-Oxybis(1-Chloropropane)	330	ug/kg
	Pyrene	330	ug/kg
	1,2,4-Trichlorobenzene	330	ug/kg
	2,4,5-Trichlorophenol	330	ug/kg
	2,4,6-Trichlorophenol	330	ug/kg
Solid Surrogates	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

MS Semivolatiles – Method 625
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Acenaphthene	10	ug/L
	Acenaphthylene	10	ug/L
	Anthracene	10	ug/L
	Benzo(a)anthracene	10	ug/L
	Benzo(b)fluoranthene	10	ug/L
	Benzo(k)fluoranthene	10	ug/L
	Benzo(ghi)perylene	10	ug/L
	Benzo(a)pyrene	10	ug/L
	bis(2-Chloroethoxy)-methane	10	ug/L
	bis(2-Chloroethyl) ether	10	ug/L
	bis(2-Chloroisopropyl) ether	10	ug/L
	bis(2-Ethylhexyl) phthalate	10	ug/L
	4-Bromophenyl phenyl ether	10	ug/L
	Butyl benzyl phthalate	10	ug/L
	4-Chloro-3-methylphenol	10	ug/L
	2-Chloronaphthalene	10	ug/L
	2-Chlorophenol	10	ug/L
	4-Chlorophenyl phenyl ether	10	ug/L
	Chrysene	10	ug/L
	Dibenz(a,h)anthracene	10	ug/L
	Di-n-butyl phthalate	10	ug/L
	1,2-Dichlorobenzene	10	ug/L
	1,3-Dichlorobenzene	10	ug/L
	1,4-Dichlorobenzene	10	ug/L
	3,3'-Dichlorobenzidine	10	ug/L
	2,4-Dichlorophenol	10	ug/L
	Diethyl phthalate	10	ug/L
	2,4-Dimethylphenol	10	ug/L
	Dimethyl phthalate	10	ug/L
	2-Methyl-4,6-dinitrophenol	50	ug/L
	2,4-Dinitrophenol	50	ug/L
	2,4-Dinitrotoluene	10	ug/L
	2,6-Dinitrotoluene	10	ug/L
	Di-n-octyl phthalate	10	ug/L
	Fluoranthene	10	ug/L
	Fluorene	10	ug/L
	Hexachlorobenzene	10	ug/L
	Hexachlorobutadiene	10	ug/L
	Hexachloroethane	10	ug/L
	Indeno(1,2,3-cd)pyrene	10	ug/L
	Isophorone	10	ug/L
	Naphthalene	10	ug/L
	Nitrobenzene	10	ug/L
	2-Nitrophenol	10	ug/L
	4-Nitrophenol	50	ug/L
	N-Nitrosodi-n-propylamine	10	ug/L
	Pentachlorophenol	10	ug/L
	Phenanthrene	10	ug/L
	Phenol	10	ug/L
	Pyrene	10	ug/L

Analyte		RL	Units
Water Surrogates	1,2,4-Trichlorobenzene	10	ug/L
	2,4,6-Trichlorophenol	10	ug/L
	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water 25 mL	Benzene	1	ug/L
	Bromobenzene	1	ug/L
	Bromochloromethane	1	ug/L
	Bromodichloromethane	1	ug/L
	Bromoform	1	ug/L
	Bromomethane	1	ug/L
	n-Butylbenzene	1	ug/L
	sec-Butylbenzene	1	ug/L
	tert-Butylbenzene	1	ug/L
	Carbon tetrachloride	1	ug/L
	Chlorobenzene	1	ug/L
	Chlorodibromomethane	1	ug/L
	Chloroethane	1	ug/L
	Chloroform	1	ug/L
	Chloromethane	1	ug/L
	2-Chlorotoluene	1	ug/L
	4-Chlorotoluene	1	ug/L
	1,2-Dibromo-3-chloropropane	2	ug/L
	1,2-Dibromoethane	1	ug/L
	Dibromomethane	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Dichlorodifluoro-methane	1	ug/L
	1,1-Dichloroethane	1	ug/L
	1,2-Dichloroethane	1	ug/L
	cis-1,2-Dichloroethene	1	ug/L
	trans-1,2-Dichloroethene	1	ug/L
	1,1-Dichloroethene	1	ug/L
	1,2-Dichloropropane	1	ug/L
	1,3-Dichloropropane	1	ug/L
	2,2-Dichloropropane	1	ug/L
	1,1-Dichloropropene	1	ug/L
	Ethylbenzene	1	ug/L
	Hexachlorobutadiene	1	ug/L
	Isopropylbenzene	1	ug/L
	p-Isopropyltoluene	1	ug/L
	Methylene chloride	1	ug/L
	Naphthalene	1	ug/L
	n-Propylbenzene	1	ug/L
	Styrene	1	ug/L
	1,1,1,2-Tetrachloroethane	1	ug/L
	1,1,2,2-Tetrachloroethane	1	ug/L
	Tetrachloroethene	1	ug/L
	Toluene	1	ug/L
	1,2,3-Trichlorobenzene	1	ug/L
	1,2,4-Trichlorobenzene	1	ug/L
	1,1,1-Trichloroethane	1	ug/L
	1,1,2-Trichloroethane	1	ug/L

Analyte	RL	Units
Trichloroethene	1	ug/L
Trichlorofluoromethane	1	ug/L
1,2,3-Trichloropropane	1	ug/L
1,2,4-Trimethylbenzene	1	ug/L
1,3,5-Trimethylbenzene	1	ug/L
Vinyl chloride	1	ug/L
m-Xylene & p-Xylene	1	ug/L
o-Xylene	1	ug/L

Water
25 mL
Surrogates

4-Bromofluorobenzene
 1,2-Dichloroethane-d4
 Toluene-d8
 Dibromofluoromethane

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water 5 mL	Benzene	5	ug/L
	Bromobenzene	5	ug/L
	Bromochloromethane	5	ug/L
	Bromodichloromethane	5	ug/L
	Bromoform	5	ug/L
	Bromomethane	5	ug/L
	n-Butylbenzene	5	ug/L
	sec-Butylbenzene	5	ug/L
	tert-Butylbenzene	5	ug/L
	Carbon tetrachloride	5	ug/L
	Chlorobenzene	5	ug/L
	Chlorodibromomethane	5	ug/L
	Chloroethane	5	ug/L
	Chloroform	5	ug/L
	Chloromethane	5	ug/L
	2-Chlorotoluene	5	ug/L
	4-Chlorotoluene	5	ug/L
	1,2-Dibromo-3-chloropropane	10	ug/L
	1,2-Dibromoethane	5	ug/L
	Dibromomethane	5	ug/L
	1,2-Dichlorobenzene	5	ug/L
	1,3-Dichlorobenzene	5	ug/L
	1,4-Dichlorobenzene	5	ug/L
	Dichlorodifluoro-methane	5	ug/L
	1,1-Dichloroethane	5	ug/L
	1,2-Dichloroethane	5	ug/L
	cis-1,2-Dichloroethene	5	ug/L
	trans-1,2-Dichloroethene	5	ug/L
	1,1-Dichloroethene	5	ug/L
	1,2-Dichloropropane	5	ug/L
	1,3-Dichloropropane	5	ug/L
	2,2-Dichloropropane	5	ug/L
	1,1-Dichloropropene	5	ug/L
	Ethylbenzene	5	ug/L
	Hexachlorobutadiene	5	ug/L
	Isopropylbenzene	5	ug/L
	p-Isopropyltoluene	5	ug/L
	Methylene chloride	5	ug/L
	Naphthalene	5	ug/L
	n-Propylbenzene	5	ug/L
	Styrene	5	ug/L
	1,1,1,2-Tetrachloroethane	5	ug/L
	1,1,2,2-Tetrachloroethane	5	ug/L
	Tetrachloroethene	5	ug/L
	Toluene	5	ug/L
	1,2,3-Trichlorobenzene	5	ug/L
	1,2,4-Trichlorobenzene	5	ug/L
	1,1,1-Trichloroethane	5	ug/L
	1,1,2-Trichloroethane	5	ug/L
	Trichloroethene	5	ug/L

Analyte	RL	Units
Trichlorofluoromethane	5	ug/L
1,2,3-Trichloropropane	5	ug/L
1,2,4-Trimethylbenzene	5	ug/L
1,3,5-Trimethylbenzene	5	ug/L
Vinyl chloride	5	ug/L
m-Xylene & p-Xylene	10	ug/L
o-Xylene	5	ug/L

Surrogates

4-Bromofluorobenzene
1,2-Dichloroethane-d4
Toluene-d8
Dibromofluoromethane

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Encore	Benzene	250	ug/kg
	Bromobenzene	250	ug/kg
	Bromochloromethane	250	ug/kg
	Bromodichloromethane	250	ug/kg
	Bromoform	250	ug/kg
	Bromomethane	250	ug/kg
	n-Butylbenzene	250	ug/kg
	sec-Butylbenzene	250	ug/kg
	tert-Butylbenzene	250	ug/kg
	Carbon tetrachloride	250	ug/kg
	Chlorobenzene	250	ug/kg
	Chlorodibromomethane	250	ug/kg
	Chloroethane	250	ug/kg
	Chloroform	250	ug/kg
	Chloromethane	250	ug/kg
	2-Chlorotoluene	250	ug/kg
	4-Chlorotoluene	250	ug/kg
	1,2-Dibromo-3-chloropropane	500	ug/kg
	1,2-Dibromoethane	250	ug/kg
	Dibromomethane	250	ug/kg
	1,2-Dichlorobenzene	250	ug/kg
	1,3-Dichlorobenzene	250	ug/kg
	1,4-Dichlorobenzene	250	ug/kg
	Dichlorodifluoro-methane	250	ug/kg
	1,1-Dichloroethane	250	ug/kg
	1,2-Dichloroethane	250	ug/kg
	cis-1,2-Dichloroethene	250	ug/kg
	trans-1,2-Dichloroethene	250	ug/kg
	1,1-Dichloroethene	250	ug/kg
	1,2-Dichloropropane	250	ug/kg
	1,3-Dichloropropane	250	ug/kg
	2,2-Dichloropropane	250	ug/kg
	1,1-Dichloropropene	250	ug/kg
	Ethylbenzene	250	ug/kg
	Hexachlorobutadiene	250	ug/kg
	Isopropylbenzene	250	ug/kg
	p-Isopropyltoluene	250	ug/kg
	Methylene chloride	250	ug/kg
	Naphthalene	250	ug/kg
	n-Propylbenzene	250	ug/kg
	Styrene	250	ug/kg
	1,1,1,2-Tetrachloroethane	250	ug/kg
	1,1,2,2-Tetrachloroethane	250	ug/kg
	Tetrachloroethene	250	ug/kg
	Toluene	250	ug/kg
	1,2,3-Trichlorobenzene	250	ug/kg
	1,2,4-Trichlorobenzene	250	ug/kg
	1,1,1-Trichloroethane	250	ug/kg
	1,1,2-Trichloroethane	250	ug/kg

	Analyte	RL	Units
	Trichloroethene	250	ug/kg
	Trichlorofluoromethane	250	ug/kg
	1,2,3-Trichloropropane	250	ug/kg
	1,2,4-Trimethylbenzene	250	ug/kg
	1,3,5-Trimethylbenzene	250	ug/kg
	Vinyl chloride	250	ug/kg
	m-Xylene & p-Xylene	500	ug/kg
	o-Xylene	250	ug/kg
Surrogates	4-Bromofluorobenzene		
	1,2-Dichloroethane-d4		
	Toluene-d8		
	Dibromofluoromethane		

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Low Level Encore	Benzene	5	ug/kg
	Bromobenzene	5	ug/kg
	Bromochloromethane	5	ug/kg
	Bromodichloromethane	5	ug/kg
	Bromoform	5	ug/kg
	Bromomethane	5	ug/kg
	n-Butylbenzene	5	ug/kg
	sec-Butylbenzene	5	ug/kg
	tert-Butylbenzene	5	ug/kg
	Carbon tetrachloride	5	ug/kg
	Chlorobenzene	5	ug/kg
	Chlorodibromomethane	5	ug/kg
	Chloroethane	5	ug/kg
	Chloroform	5	ug/kg
	Chloromethane	5	ug/kg
	2-Chlorotoluene	5	ug/kg
	4-Chlorotoluene	5	ug/kg
	1,2-Dibromo-3-chloropropane	10	ug/kg
	1,2-Dibromoethane	5	ug/kg
	Dibromomethane	5	ug/kg
	1,2-Dichlorobenzene	5	ug/kg
	1,3-Dichlorobenzene	5	ug/kg
	1,4-Dichlorobenzene	5	ug/kg
	Dichlorodifluoro-methane	5	ug/kg
	1,1-Dichloroethane	5	ug/kg
	1,2-Dichloroethane	5	ug/kg
	cis-1,2-Dichloroethene	5	ug/kg
	trans-1,2-Dichloroethene	5	ug/kg
	1,1-Dichloroethene	5	ug/kg
	1,2-Dichloropropane	5	ug/kg
	1,3-Dichloropropane	5	ug/kg
	2,2-Dichloropropane	5	ug/kg
	1,1-Dichloropropene	5	ug/kg
	Ethylbenzene	5	ug/kg
	Hexachlorobutadiene	5	ug/kg
	Isopropylbenzene	5	ug/kg
	p-Isopropyltoluene	5	ug/kg
	Methylene chloride	5	ug/kg
	Naphthalene	5	ug/kg
	n-Propylbenzene	5	ug/kg
	Styrene	5	ug/kg
	1,1,1,2-Tetrachloroethane	5	ug/kg
	1,1,2,2-Tetrachloroethane	5	ug/kg
	Tetrachloroethene	5	ug/kg
	Toluene	5	ug/kg
	1,2,3-Trichlorobenzene	5	ug/kg
	1,2,4-Trichlorobenzene	5	ug/kg
	1,1,1-Trichloroethane	5	ug/kg
	1,1,2-Trichloroethane	5	ug/kg
	Trichloroethene	5	ug/kg

	Analyte	RL	Units
	Trichlorofluoromethane	5	ug/kg
	1,2,3-Trichloropropane	5	ug/kg
	1,2,4-Trimethylbenzene	5	ug/kg
	1,3,5-Trimethylbenzene	5	ug/kg
	Vinyl chloride	5	ug/kg
	m-Xylene & p-Xylene	10	ug/kg
	o-Xylene	5	ug/kg

Surrogates	4-Bromofluorobenzene
	1,2-Dichloroethane-d4
	Toluene-d8
	Dibromofluoromethane

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Frozen Encore	Benzene	5	ug/kg
	Bromobenzene	5	ug/kg
	Bromochloromethane	5	ug/kg
	Bromodichloromethane	5	ug/kg
	Bromoform	5	ug/kg
	Bromomethane	5	ug/kg
	n-Butylbenzene	5	ug/kg
	sec-Butylbenzene	5	ug/kg
	tert-Butylbenzene	5	ug/kg
	Carbon tetrachloride	5	ug/kg
	Chlorobenzene	5	ug/kg
	Chlorodibromomethane	5	ug/kg
	Chloroethane	5	ug/kg
	Chloroform	5	ug/kg
	Chloromethane	5	ug/kg
	2-Chlorotoluene	5	ug/kg
	4-Chlorotoluene	5	ug/kg
	1,2-Dibromo-3-chloropropane	10	ug/kg
	1,2-Dibromoethane	5	ug/kg
	Dibromomethane	5	ug/kg
	1,2-Dichlorobenzene	5	ug/kg
	1,3-Dichlorobenzene	5	ug/kg
	1,4-Dichlorobenzene	5	ug/kg
	Dichlorodifluoro-methane	5	ug/kg
	1,1-Dichloroethane	5	ug/kg
	1,2-Dichloroethane	5	ug/kg
	cis-1,2-Dichloroethene	5	ug/kg
	trans-1,2-Dichloroethene	5	ug/kg
	1,1-Dichloroethene	5	ug/kg
	1,2-Dichloropropane	5	ug/kg
	1,3-Dichloropropane	5	ug/kg
	2,2-Dichloropropane	5	ug/kg
	1,1-Dichloropropene	5	ug/kg
	Ethylbenzene	5	ug/kg
	Hexachlorobutadiene	5	ug/kg
	Isopropylbenzene	5	ug/kg
	p-Isopropyltoluene	5	ug/kg
	Methylene chloride	5	ug/kg
	Naphthalene	5	ug/kg
	n-Propylbenzene	5	ug/kg
	Styrene	5	ug/kg
	1,1,1,2-Tetrachloroethane	5	ug/kg
	1,1,2,2-Tetrachloroethane	5	ug/kg
	Tetrachloroethene	5	ug/kg
	Toluene	5	ug/kg
	1,2,3-Trichlorobenzene	5	ug/kg
	1,2,4-Trichlorobenzene	5	ug/kg
	1,1,1-Trichloroethane	5	ug/kg
	1,1,2-Trichloroethane	5	ug/kg
	Trichloroethene	5	ug/kg

	Analyte	RL	Units
	Trichlorofluoromethane	5	ug/kg
	1,2,3-Trichloropropane	5	ug/kg
	1,2,4-Trimethylbenzene	5	ug/kg
	1,3,5-Trimethylbenzene	5	ug/kg
	Vinyl chloride	5	ug/kg
	m-Xylene & p-Xylene	10	ug/kg
	o-Xylene	5	ug/kg
Surrogates	4-Bromofluorobenzene		
	1,2-Dichloroethane-d4		
	Toluene-d8		
	Dibromofluoromethane		

MS Volatiles – Method 624
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Benzene	5	ug/L
	Bromodichloro-methane	5	ug/L
	Bromoform	5	ug/L
	Bromomethane	55	ug/L
	Carbon tetrachloride	5	ug/L
	Chlorobenzene	5	ug/L
	Dibromochloro-methane	5	ug/L
	Chloroethane	5	ug/L
	2-Chloroethyl vinyl ether	--	ug/L
	Chloroform	5	ug/L
	Chloromethane	5	ug/L
	1,2-Dichlorobenzene	5	ug/L
	1,3-Dichlorobenzene	5	ug/L
	1,4-Dichlorobenzene	5	ug/L
	1,1-Dichloroethane	5	ug/L
	1,2-Dichloroethane	5	ug/L
	trans-1,2-Dichloroethene	5	ug/L
	1,1-Dichloroethene	5	ug/L
	1,2-Dichloropropane	5	ug/L
	cis-1,3-Dichloropropene	5	ug/L
	trans-1,3-Dichloropropene	5	ug/L
	Ethylbenzene	5	ug/L
	Methylene chloride	5	ug/L
	1,1,2,2-Tetrachloroethane	5	ug/L
	Tetrachloroethene	5	ug/L
	Toluene	5	ug/L
	1,1,1-Trichloroethane	5	ug/L
	1,1,2-Trichloroethane	5	ug/L
	Trichloroethene	5	ug/L
	Trichlorofluoro-methane	5	ug/L
	Vinyl chloride	5	ug/L
Surrogates	Bromofluorobenzene		
	1,2-Dichloro-ethane-d4		
	Toluene-d8		

GC Semivolatiles – Method 8081A
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aldrin	0.05	ug/L
	alpha-BHC	0.05	ug/L
	beta-BHC	0.05	ug/L
	delta-BHC	0.05	ug/L
	gamma-BHC (Lindane)	0.05	ug/L
	alpha-Chlordane	0.05	ug/L
	gamma-Chlordane	0.05	ug/L
	4,4'-DDD	0.05	ug/L
	4,4'-DDE	0.05	ug/L
	4,4'-DDT	0.05	ug/L
	Dieldrin	0.05	ug/L
	Endosulfan I	0.05	ug/L
	Endosulfan II	0.05	ug/L
	Endosulfan sulfate	0.05	ug/L
	Endrin	0.05	ug/L
	Endrin aldehyde	0.05	ug/L
	Endrin ketone	0.05	ug/L
	Heptachlor	0.05	ug/L
	Heptachlor epoxide	0.05	ug/L
	Methoxychlor	0.1	ug/L
	Toxaphene	2	ug/L
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Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

GC Semivolatiles – 8081A
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Solid	Aldrin	1.7	ug/kg
	alpha-BHC	1.7	ug/kg
	beta-BHC	1.7	ug/kg
	delta-BHC	1.7	ug/kg
	gamma-BHC (Lindane)	1.7	ug/kg
	alpha-Chlordane	1.7	ug/kg
	gamma-Chlordane	1.7	ug/kg
	4,4'-DDD	1.7	ug/kg
	4,4'-DDE	1.7	ug/kg
	4,4'-DDT	1.7	ug/kg
	Dieldrin	1.7	ug/kg
	Endosulfan I	1.7	ug/kg
	Endosulfan II	1.7	ug/kg
	Endosulfan sulfate	1.7	ug/kg
	Endrin	1.7	ug/kg
	Endrin aldehyde	1.7	ug/kg
	Endrin ketone	1.7	ug/kg
	Heptachlor	1.7	ug/kg
	Heptachlor epoxide	1.7	ug/kg
	Methoxychlor	3.3	ug/kg
	Toxaphene	67	ug/kg
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Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

GC Semivolatiles – Method 8082
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aroclor 1016	1	ug/L
	Aroclor 1221	1	ug/L
	Aroclor 1232	1	ug/L
	Aroclor 1242	1	ug/L
	Aroclor 1248	1	ug/L
	Aroclor 1254	1	ug/L
	Aroclor 1260	1	ug/L
	Aroclor 1262	1	ug/L
	Aroclor 1268	1	ug/L
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Surrogates	Decachlorobi-phenyl Tetrachloro-m-xylene		
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Solid	Aroclor 1016	33	ug/kg
	Aroclor 1221	33	ug/kg
	Aroclor 1232	33	ug/kg
	Aroclor 1242	33	ug/kg
	Aroclor 1248	33	ug/kg
	Aroclor 1254	33	ug/kg
	Aroclor 1260	33	ug/kg
	Aroclor 1262	33	ug/kg
	Aroclor 1268	33	ug/kg
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Surrogates	Decachloro-biphenyl Tetrachloro-m-xylene		

GC Semivolatiles – Method 608
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aldrin	0.05	ug/L
	alpha-BHC	0.05	ug/L
	beta-BHC	0.05	ug/L
	delta-BHC	0.05	ug/L
	gamma-BHC (Lindane)	0.05	ug/L
	Chlordane (technical)	0.5	ug/L
	4,4'-DDD	0.05	ug/L
	4,4'-DDE	0.05	ug/L
	4,4'-DDT	0.05	ug/L
	Dieldrin	0.05	ug/L
	Endosulfan I	0.05	ug/L
	Endosulfan II	0.05	ug/L
	Endosulfan sulfate	0.05	ug/L
	Endrin	0.05	ug/L
	Endrin aldehyde	0.05	ug/L
	Heptachlor	0.05	ug/L
	Heptachlor epoxide	0.05	ug/L
	PCB-1016	1	ug/L
	PCB-1221	1	ug/L
	PCB-1232	1	ug/L
	PCB-1242	1	ug/L
	PCB-1248	1	ug/L
	PCB-1254	1	ug/L
	PCB-1260	1	ug/L
	Toxaphene	2	ug/L
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Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

GC Semivolatiles – Method 8151A
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	2,4-D	4	ug/L
	Dalapon	2	ug/L
	2,4-DB	4	ug/L
	Dicamba	2	ug/L
	Dichlorprop	4	ug/L
	Dinoseb	0.6	ug/L
	MCPA	400	ug/L
	MCPP	400	ug/L
	2,4,5-TP (Silvex)	1	ug/L
	2,4,5-T	1	ug/L
Surrogate	2,4-Dichlorophenylacetic acid		
Solid	2,4-D	80	ug/kg
	Dalapon	40	ug/kg
	2,4-DB	80	ug/kg
	Dicamba	40	ug/kg
	Dichlorprop	80	ug/kg
	Dinoseb	12	ug/kg
	MCPA	8000	ug/kg
	MCPP	8000	ug/kg
	2,4,5-TP (Silvex)	20	ug/kg
	2,4,5-T	20	ug/kg
Surrogate	2,4-Dichlorophenylacetic acid		

GC Volatiles - Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

		Analyte	RL	Units
Water 5 mL Purge		Benzene	1	ug/L
		Bromobenzene	1	ug/L
		Bromochloromethane	1	ug/L
		Bromodichloromethane	1	ug/L
		Bromoform	1	ug/L
		Bromomethane	1	ug/L
		n-Butylbenzene	1	ug/L
		sec-Butylbenzene	1	ug/L
		tert-Butylbenzene	1	ug/L
		Carbon tetrachloride	1	ug/L
		Chlorobenzene	1	ug/L
		Chlorodibromomethane	1	ug/L
		Chloroethane	1	ug/L
		Chloroform	1	ug/L
		Chloromethane	1	ug/L
		2-Chlorotoluene	1	ug/L
		4-Chlorotoluene	1	ug/L
		1,2-Dibromo-3-chloropropane (DBCP)	1	ug/L
		1,2-Dibromoethane (EDB)	1	ug/L
		Dibromomethane	1	ug/L
		1,2-Dichlorobenzene	1	ug/L
		1,3-Dichlorobenzene	1	ug/L
		1,4-Dichlorobenzene	1	ug/L
		Dichlorodifluoromethane	1	ug/L
		1,1-Dichloroethane	1	ug/L
		1,2-Dichloroethane	1	ug/L
		cis-1,2-Dichloroethene	1	ug/L
		trans-1,2-Dichloroethene	1	ug/L
		1,1-Dichloroethene	1	ug/L
		1,2-Dichloropropane	1	ug/L
		1,3-Dichloropropane	1	ug/L
		2,2-Dichloropropane	1	ug/L
		cis-1,3-Dichloropropene	1	ug/L
		trans-1,3-Dichloropropene	1	ug/L
		1,1-Dichloropropene	1	ug/L
		Ethylbenzene	1	ug/
		Hexachlorobutadiene	1	ug/L
		Isopropylbenzene	1	ug/L
		p-Isopropyltoluene	1	ug/L
		Methylene chloride	5	ug/L
		Naphthalene	1	ug/L
		n-Propylbenzene	1	ug/L
		Styrene	1	ug/L
		1,1,1,2-Tetrachloroethane	1	ug/L
		1,1,2,2-Tetrachloroethane	1	ug/L
		Tetrachloroethene	1	ug/L
		Toluene	1	ug/L
		1,2,3-Trichlorobenzene	1	ug/L
		1,2,4-Trichlorobenzene	1	ug/L

GC Volatiles - Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

Analyte		RL	Units
1,1,1-Trichloroethane		1	ug/L
1,1,2-Trichloroethane		1	ug/L
Trichloroethene		1	ug/L
Trichlorofluoromethane		1	ug/L
1,2,3-Trichloropropane		1	ug/L
1,2,4-Trimethylbenzene		1	ug/L
1,3,5-Trimethylbenzene		1	ug/L
Vinyl chloride		1	ug/L
m-Xylene & p-Xylene		2	ug/L
o-Xylene		1	ug/L
Xylenes (total)		3	ug/L
<hr/>			
Surrogates	1,4-Dichlorobutane		
	Trifluorotoluene		

GC Volatiles - Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

		Analyte	RL	Units
Solid 5 g		Benzene	1	ug/kg
		Ethylbenzene	1	ug/kg
		Methyl tert-butyl ether (MTBE)	1	ug/kg
		Methyl tert-butyl ether	1	ug/kg
		Naphthalene	1	ug/kg
		Toluene	1	ug/kg
		1,2,4-Trimethylbenzene	1	ug/kg
		1,3,5-Trimethylbenzene	1	ug/kg
		m-Xylene & p-Xylene	2	ug/kg
		o-Xylene	1	ug/kg
		Xylenes (total)	3	ug/kg
Surrogates		Trifluorotoluene		

GC Volatiles – Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Solid Encore, Methanol Prep	Benzene	50	ug/kg
	Ethylbenzene	50	ug/kg
	Methyl tert-butyl ether (MTBE)	50	ug/kg
	Naphthalene	250	ug/kg
	Toluene	50	ug/kg
	1,2,4-Trimethylbenzene	50	ug/kg
	1,3,5-Trimethylbenzene	50	ug/kg
	m-Xylene & p-Xylene	100	ug/kg
	o-Xylene	50	ug/kg
	Xylenes (total)	150	ug/kg
Surrogates	Trifluorotoluene		

GC Volatiles – Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Frozen Encore	Benzene	1	ug/kg
	Ethylbenzene	1	ug/kg
	Methyl tert-butyl ether (MTBE)	1	ug/kg
	Methyl tert-butyl ether	1	ug/kg
	Naphthalene	1	ug/kg
	Toluene	1	ug/kg
	1,2,4-Trimethylbenzene	1	ug/kg
	1,3,5-Trimethylbenzene	1	ug/kg
	m-Xylene & p-Xylene	2	ug/kg
	o-Xylene	1	ug/kg
	Xylenes (total)	3	ug/kg

Surrogates	Trifluorotoluene
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GC Volatiles – Method 601
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Bromodichloromethane	1	ug/L
	Bromoform	1	ug/L
	Bromomethane	1	ug/L
	Carbon tetrachloride	1	ug/L
	Chlorobenzene	1	ug/L
	Dibromochloromethane	1	ug/L
	Chloroethane	1	ug/L
	2-Chloroethyl vinyl ether	5	ug/L
	Chloroform	1	ug/L
	Chloromethane	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Dichlorodifluoromethane	1	ug/L
	1,1-Dichloroethane	1	ug/L
	1,2-Dichloroethane	1	ug/L
	trans-1,2-Dichloroethene	1	ug/L
	1,1-Dichloroethene	1	ug/L
	1,2-Dichloropropane	1	ug/L
	cis-1,3-Dichloropropene	1	ug/L
	trans-1,3-Dichloropropene	1	ug/L
	Methylene chloride	5	ug/L
	1,1,2,2-Tetrachloroethane	1	ug/L
	Tetrachloroethene	1	ug/L
	1,1,1-Trichloroethane	1	ug/L
	1,1,2-Trichloroethane	1	ug/L
	Trichloroethene	1	ug/L
	Trichlorofluoromethane	1	ug/L
	Vinyl chloride	1	ug/L
<hr/>			
Surrogates	1,4-Dichlorobutane		

GC Volatiles – Method 602
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Benzene	1	ug/L
	Chlorobenzene	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Ethylbenzene	1	ug/L
	Toluene	1	ug/L
<hr/>			
Surrogates	Trifluorotoluene		

**Total Petroleum Hydrocarbons, Diesel & Gasoline Range Organics
Reporting Limits (RL), Method Reference ^{1,2}**

	Analyte	RL	Units
Diesel Range Organics - Water	TPH (as Diesel)	100	ug/L
Surrogate	C9 (nonane)		
Diesel Range Organics - Solid	TPH (as Diesel)	10	mg/kg
Surrogate	C9 (nonane)		
Gasoline Range Organics - Water	Gasoline Range Organics	100	ug/L
	TPH (as Gasoline)	100	ug/L
	TPH (Purgeables)	100	ug/L
Gasoline Range Organics - Solid	Gasoline Range Organics	100	ug/kg
	TPH (as Gasoline)	100	ug/kg
	TPH (Purgeables)	100	ug/kg
Surrogate	TFT		
Gasoline Range Organics – Medium Level	TPH (as Gasoline) Solid	1000	ug/kg
	TPH (as Gasoline)	1000	ug/kg
	TPH (Purgeables)	1000	ug/kg
Surrogate	TFT		

Footnote:

¹ The latest MDLs, RLs, and Control Limits will be utilized at the time of sample analysis, and are available upon request.

² The compounds listed represent TAL/TCL compounds. Project specific or other regulatory lists may be available upon request.

Appendix 6. Glossary/Acronyms

Glossary

Acceptance Criteria:

Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority:

The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

Accuracy:

The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst:

The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Assessment:

The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

Assessment Criteria:

The measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Assessment Team:

The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

Assessor:

One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)

Audit:

A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch:

Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank:

A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample:

A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:

To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve:

The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:

A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:

A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):

A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30-2.2)

Chain-of-Custody:

An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:

The enabling legislation in 42 U.S.C. 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):

The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

Compromised Samples:

Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

Confidential Business Information (CBI):

Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:

Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

- Second column confirmation
- Alternate wavelength
- Derivatization
- Mass spectral interpretation
- Alternative detectors or
- Additional Cleanup procedures

(NELAC)

Conformance:

An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Corrective Action:

The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit:

A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction:

The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency:

An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Detection Limit:

The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control:

The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicate Analyses:

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Environmental Detection Limit (EDL):

The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

Equipment Blank:

Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

External Standard Calibration:

Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):

The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

Federal Water Pollution Control Act (Clean Water Act, CWA):

The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:

Blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Testing:

NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Finding:

An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

Holding Times (Maximum Allowable Holding Times):

The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Inspection:

An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard:

A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration:

Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:

A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Response:

Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

Laboratory:

A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):

A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:

Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1st Order Curve):

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):

An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Manager (however named):

The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

Matrix:

The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with .15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with .15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:

A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same

conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit:

The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

National Environmental Laboratory Accreditation Conference (NELAC):

A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP):

The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control:

Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

NELAC Standards:

The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)

Performance Audit:

The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS):

A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control:

Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:

The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation:

Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing:

A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program:

The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT):

A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Quality Assurance:

An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP):

A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:

The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:

An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual:

A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System:

A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The

quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

Quantitation Limits:

The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range:

The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Reagent Blank (method reagent blank):

A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reference Material:

A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:

A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard:

A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Replicate Analyses:

The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement:

Denotes a mandatory specification; often designated by the term "shall". (NELAC)

Resource Conservation and Recovery Act (RCRA):

The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Safe Drinking Water Act (SDWA):

The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Duplicate:

Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity:

(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity:

The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike:

A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period. (NELAC)

Standard:

The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):

A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM):

A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named):

The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate:

A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):

A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director:

Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test:

A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:

An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Toxic Substances Control Act (TSCA):

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:

The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Uncertainty:

A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

United States Environmental Protection Agency (EPA):

The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:

The process of substantiating specified performance criteria. (EPA-QAD)

Verification:

Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell:

A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Acronyms

BS – Blank Spike
BSD – Blank Spike Duplicate
CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
CRS – Change Request Form
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DU – Duplicate
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
MDL – Method Detection Limit
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAC - National Environmental Laboratory Accreditation Conference
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 7.

Laboratory Certifications, Accreditations, Validations

TestAmerica North Canton maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Certificate Number	Organization	Certificate Number
California	01144CA	New Jersey	OH001
Connecticut	PH-0590	New York	10975
Florida	E87225	Pennsylvania	68-00340
Georgia	--	OVAP	CL0024
Illinois	200004	US ACE (Army)	--
Kansas	E-10336	USDA (Dept.of Agriculture)	S-62345
Kentucky Underground Storage Tank Program	0058	West Virginia	210
Minnesota	039-999-348	Wisconsin	999518190

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

Claims of Accreditation Status

TestAmerica North Canton has agreed to make only valid claims as to its accreditation/certification status by any authority by ensuring that the expiration dates are not exceeded and the method-specific scope or parameter lists are supportable, as required by each. Any false claims would be reported to that authority. The agreement covers the use of the authority's name, such as "Authority-Accredited," logo, or certificate number. The only valid proof of accreditation/certification is the current certificate and scope of the authority. It is the responsibility of the laboratory to make these documents available to all staff, and it is the staff's duty to reference only the current documents.

A report with scope and non-scope analytes may only be presented on the same report if the non-accredited results are clearly and unambiguously identified. No report with non-scope analytes may be associated with the logo, "Authority accredited" phrase, or the certificate number. Only the analytes specified by a unique method are valid within the scope. There shall be no intentional misleading of the users of the laboratory's services in this regard.

No opinions and/or interpretations based on results outside the laboratory's scope may be presented on a document referenced by "Authority-accredited, the logo, or the certificate number. If these are made, they must be written in a separate letter which is not endorsed by the authority.

The "Authority-accredited" logo may only be affixed to equipment calibrated by a laboratory that is accredited by the authority. If calibration labels contain the logo, they must also show the calibration laboratory's name or its certificate number, the instrument's unique identification, the date of the last calibration, and a cross-reference to the last calibration certificate.

Should the company decide to use the "Authority-accredited" logo in marketing activities, no misrepresentation may occur. Only reference to the accredited scope at a specific laboratory site is allowed. If any "Authority-accredited" language is used in proposals or quotations, any non-scope analytes must be clearly denoted as not accredited by that authority. The same is true for any use of laboratory letterhead with the "Authority-accredited" wording or logo. The logo may not be affixed to any material, item, product, part, or packaging, thereby implying accreditation status to that piece. In literature, any use of the logo must be positioned adjacent to the accredited laboratory's name and clearly state that the presence of the logo does not imply certification/approval of the products tested. At no time may the logo appear to suggest that a person is accredited. Misrepresentation of accreditation status is never allowed and must be reported if it occurs. If in doubt, the idea of the logo's use may be presented to the authority for approval.

If accreditation is terminated or suspended, the laboratory will immediately cease to use the "Authority-accredited" wording, the logo, or the certificate number reference in any way and inform clients impacted by the change.

Appendix 8.

Data Qualifiers

Qualifier Organic	Qualifier Inorganic	Footnote
U	U	Analyte analyzed for but was not detected.
G	G	Elevated reporting limit. The reporting limit is elevated due to matrix interference.
J	B	Estimated result. Result is less than RL.
E	I	Estimated result. Result concentration exceeds the calibration range.
M	M	Matrix spike recovery outside of limits.
B	J	Method blank contamination. The associated method blank contains the target analyte at a reportable level.
P	*	Relative percent difference (RPD) is outside stated control limits.
a	N	Spiked analyte recovery is outside stated control limits.
*		Surrogate recovery is outside stated control limits.
PG		The percent difference between the original and confirmation analyses is greater than 40%.

This is not an exhaustive list of qualifiers. All qualifiers are defined on each data sheet. Client specific qualifiers may also be used, and would also be defined on the data sheet.

TestAmerica North Canton

Facility Addendum And Contingency Plan

Revised Deceber, 2007

Approvals and Signatures:

Laboratory Director: _____ Date: December 15th, 2007
Opal Davis-Johnson

Environmental Health
and Safety Coordinator: _____ Date: December 15th, 2007
Stephen J Jackson

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TestAmerica North Canton Facility Addendum

1.0 Scope and Purpose

The purpose of this document is to provide guidelines and procedures for the implementation of the TestAmerica Corporate Health and Safety Manual at the North Canton Laboratory.

Persons responsible for Health and Safety are listed on the Emergency Contact list included in Appendix A to this document. This list is prominently posted in all departments of the laboratory and throughout the building. **If any employee disregards this policy he or she will be subject to TestAmerica disciplinary actions.**

2.0 Building Security

To provide for the safety and security of employees and the facility, only authorized visitors are allowed in the workplace. Restricting unauthorized visitors helps maintain safety standards, protects against theft, ensures security of equipment, protects confidential information, safeguards employee welfare, and avoids potential distractions and disturbances.

All employees are issued a security key to be used at the patio, side and back doors. These doors should never be propped open unless actively loading or unloading.

All authorized visitors must enter through the main entrance and sign in at the front desk to receive a visitor's badge.

Authorized visitors will receive directions or be escorted to their destination.

All authorized visitors must adhere to the PPE requirements for the area(s), in which they are conducting business.

If an unauthorized individual is observed on TestAmerica's premise, employees should immediately notify their supervisor or, if necessary direct the individual to the main entrance.

North Canton's business hours are 8:00 - 5:00 on Monday through Friday and 9:00 -12:00 on Saturday. Samples are not to be accepted outside business hours unless the laboratory has been notified in advance by the Project Manager.

3.0 Emergency Procedures

Emergency situation that could occur at North Canton includes fires, spills, severe weather, power failures, terrorist threats and fume hood failure. Procedures specific to North Canton are listed in this section of the Facility Addendum. Any procedures not listed in this section can be found in Chapter 7 of the Corporate Safety Manual. **3.1 Fires**

3.1.1 Procedures for Responding to Fires

In the event a fire occurs in an area where an employee is working, the employee must:

- A. Evacuate the immediate area. Direct others away from the fire and to the nearest exit.
- B. Notify all employees in the facility that there is a fire, no matter how small. Notification can be made over the PA system by dialing 88 or using the Page button. Pulling one of the fire alarm pull stations, located near the exits to the building will trigger an audible alarm that can be heard throughout the facility and at the Jackson Township Fire Department.

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Protec security will contact Maintenance, the Laboratory Director and the EH&S Coordinator in the event that the alarm sounds after normal business hours.

- C. Contact the Jackson Township Fire Department by calling 9-911 or by pulling the fire alarm. When speaking to the fire department, always relay the company name and exact location of the fire within the facility.

TestAmerica North Canton
4101 Shuffel Street NW
North Canton, Ohio 44720

- D. Confine the fire to a specific area by closing doors.

Only trained employees can use a fire extinguisher to attempt to put the fire out.

- E. After containing the fire, the employee should evacuate the area and report to their Staging Area.

3.1.2 Use of Fire Extinguishers

- A. If trained, employees may use the nearest fire extinguisher and attempt to extinguish the fire. Prior to doing this, the trained employee must assess the fire to ensure that it is small, contained, exits are open, that the extinguisher is of the proper type and that there is an available escape route. A second employee, or buddy, must be nearby to assist.
- B. After attempting to extinguish the fire, the employee must immediately evacuate the facility and move to their designated staging area.

3.1.3 Response to Fire Alarm

- A. In the event that the fire alarm sounds, all personnel within the facility should:
- Quickly end phone conversations.
 - If there is sufficient time, turn off all instrumentation.
 - Immediately leave the building by the assigned evacuation routes.
 - Congregate in the assigned staging area in the front parking lot.
- B. Personnel operating open flames, hot plates, or heating mantles should conduct an orderly shut down as they exit. Evacuation routes are posted in each department, as is the staging area location. These documents are located near exits.
- C. Employees that are in an unfamiliar area of the facility during a fire drill will follow other personnel to the nearest emergency exit, and report to their assigned staging area once outside for roll call. Personnel waiting in the staging areas must not block any entries for emergency vehicles and must keep fire lanes clear at all times.
- D. Personnel may not re-enter the facility following an alarm until the problem has been identified and permission to re-enter given by the laboratory director, EH&S coordinator, or a fire department officer.
- E. The receptionist will bring the visitor log to the mailbox located near the southeast corner and will assist in determining that all visitors are accounted for.

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3.1.4 Fire Captains and designated sweep areas

- A. Individuals within the facility have been designated as fire captains. These individuals are listed in Appendix B of this document. The fire captains will work in pairs and will pass through assigned sections of the facility to ensure that all personnel have been evacuated, all doors have been closed and hoods turned off and that instruments have been adequately shut down.
- B. The fire captains will congregate at the mailbox located near the southeast corner of the facility following the sweep of their area.
- C. The LD, Opps manager, Maintenance, and EH&S will assemble at the mailbox to determine any imposing hazards that may need assessed such as severity of the situation, evacuation of other business's and assist Jackson Fire Department with any needs or information.

Fire Captain Responsibilities

- Notify personnel in sweep areas of fire and evacuate.
- Ensure that all persons are evacuated from areas.
- Ensure that all doors and hoods are closed.
- Exit the building.
- Fire Captains will then communicate between the staging area leaders, the Lab Director and EH&S on roll call of personnel.

3.1.5 Staging Area Leaders

Staging Area Leaders are responsible for checking to see that all members of their group have been accounted for and alerting the fire captains if an employee is missing and feared to be in the building.

Each staging leader will get a copy of the roster for their respective staging area and flags (red, yellow and green) printed with the number of their staging area. The staging area leader will use the roster to take a roll call. The flags will be used to indicate the status of the roll call: red indicates someone is unaccounted for; yellow indicates that the roll call is in progress and green indicates that everyone is accounted for. The use of the flags will allow the LD and fire captains to quickly assess the status of each staging area. These items will be obtained by all major exits.

3.1.6 Apparent false alarms

In cases where the alarm sounds with no apparent fire, employees are to follow all steps listed above. The alarm is equipped with a digital panel that indicates which pull station has been activated. Assuming no fire is detected during the sweep of the building and the alarm readout indicates a false alarm, Protec security can be called to stop the fire department's arrival. If there is any doubt as to the cause of the alarm, or if the alarm sounds on second shift, employees should wait for the fire department and allow them to determine that there is no fire.

3.2 Spill Response

All spills must be handled safely. If there is any doubt about the proper procedures, or the proper size category of the spill, evacuate immediately and contact the Waste Coordinator, EH&S or an area supervisor. The TestAmerica North Canton Laboratory does not have an emergency spill response team on site, and relies on the Jackson Township fire department for clean up of large

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spills. Details of the TestAmerica policies for handling small spills of hazardous or non-hazardous materials are located in section 7.9 of the TestAmerica Corporate Safety Manual.

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All employees are trained to clean up small spills of hazardous materials that occur in their work area. The term “small” is relative when it comes to deciding what an employee can, or cannot clean up without assistance. A small spill of an extremely toxic material, outside of a fume hood or properly ventilated area may be less than 50 milliliters. On the other hand, if the same material is contained in a fume hood or properly ventilated area, anything less than 500 ml would be considered small. Likewise, an employee could clean up several gallons of a relatively non-toxic material.

Consult Tables 7-2, 7-3, 7-4 and 7-5 in section 7.9.1 of the CSM, to determine the appropriate category of a spill (small vs. large). **Quantities listed can be multiplied by 10 if the spill occurs in a well ventilated area, such as a hood.**

If the spill qualifies as a large spill, request help from the Jackson Fire Department.

Always consult Material Safety Data Sheets (MSDS's) before attempting to clean up any spill. Each employee is expected to use their best judgment when determining whether they can safely clean up a spill. A review of the appropriate MSDS will aid in this decision.

3.2.1 Procedure for responding to small spills:

If a spill of a material is small and non-threatening, employees will:

- Alert employees in the immediate area that there has been a spill.
- Turn off all ignition sources.
- Clean up the spill. Employees will use personal protective equipment (PPE) appropriate to that chemical when cleaning up the spilled material.
- Notify Waste Coordinator, EH&S or area supervisor that a material has been spilled.
- If the spilled material could have caused injury to employees or damaged property, the EH&S must be notified.
- If appropriate, complete an incident or near miss report for EH&S.

3.2.3 Procedures for responding to Large spills:

Some large spills can be handled without taking unusual precautions. For instance, water samples that are spilled can normally be cleaned up without special measures. Some large spills will require special precautions.

If the amount of material spilled is large, the employees will:

- Get everyone out of the immediate area. Large spills not contained in a room will require the evacuation of the entire building.
- After checking with EH&S, activate the emergency ventilation system by pushing the emergency ventilation button (large and red) located in the hallways outside of the downstairs labs or by the electrical panel.
- Secure all entrances so people cannot go into the affected area.
- Contact the Waste Coordinator, EH&S and the Lab Manager. They will assess the situation a safe distance from the spill and determine the appropriate response. Appropriate actions can range from TestAmerica employees cleaning up the spill, to summoning the Jackson Township Fire Department. The Waste Coordinator, EH&S and the Lab Manager will determine whether the other businesses in the complex need to be notified.
- Assist in assessing the severity of the event, gathering information about the spilled chemical and in determining that all employees are accounted for and out of the building.
- Wait for further instructions. The Waste Coordinator or EH&S Coordinator will decide when it is safe to return to a work area.

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In the event of a large spill where the individuals listed above cannot be contacted, the TestAmerica Corporate EH&S should be notified.

Determining Appropriate Cleanup Actions

In the event of a large spill, the Waste Coordinator and EH&S Coordinator will determine the actions to be taken to clean up the spill. MSDSs must be consulted in making this decision.

If a volatile solvent has been spilled, the chemical will be allowed to evaporate. Hoods should be left on, however make-up air should be turned off. The approximate time to clear the room of the solvent vapors can be calculated from the approximate amount of material spilled, estimated airflow changes in the area, and appropriate evaporation rate. Air quality should be tested using Drager tubes prior to re-opening an area.

In the event that large volumes of acids or other non-volatiles are spilled, the Jackson Township Fire Department should be contacted. After hours, the waste coordinator, Laboratory Director or EH&S should be called in that order.

Drager Tube Sampling

1. Drager tubes and all necessary supplies (air sampling tubes), sample collector, and tubing) can be found in the Waste Coordinator's office
2. A tube appropriate for the spilled material should be chosen. Instructions are available in the office and should be reviewed prior to sampling.
3. The air sample tube is placed in the tubing then connected to the sample collector. The tube can be slid under the door of the closed off area while sampling the air to further ensure that no employees will be exposed.
4. The tubes are compared to the color chart provided with the sampling kits to determine if there are measurable concentrations of the chemical

The Waste Coordinator, EH&S and the Lab Manager will decide when the area can be re-entered.

Spill Containment

The North Canton laboratory does not have floor drains, which could allow spills to reach the sewer system. In the event a spill occurs outside of the laboratory, such as when waste is being transported to the waste building, barriers must be immediately placed around the sewer entrances to prevent the material from entering the sewer system. Proper barriers can be found in the waste building.

Spill Kits can be found in the following locations

- Small spill kits are located in each lab.
- Mounted spill cabinets on both ends of the north/south hallway going toward the warehouse
- First east/west hall way of the main lab (bullpen) area
- Waste building – includes barrier controls

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The Spill Response Cart is located in the hall across from the TCLP extraction room.

Spill Cart Contents:

- Chemical suits
- Gloves
- Eye protection
- Disposal bags
- Clean-up materials

Material Safety Data Sheets (MSDS's) are located on the home page of Oasis, or on a back up CD located in IT.

3.3 Accidents and Exposures

Accident and Exposure procedures can be found in the Corporate Safety Manual. Accident procedures can be found in Section 7.5 and procedures for handling exposures are found in Section 7.7.

All accidents, illnesses and near misses must be reported. Reports are to be made on the TestAmerica Accident Forms and are given to the Group Leader, EH& S or Lab Director. Accidents must be reported as soon as possible following appropriate resolution of the event. Accident forms can be found at every First Aid station and online at the EH&S site on Oasis. Accident reports are to be submitted to the corporate EH&S electronically.

A minor injury is one that is not life-threatening and does not involve severe danger or damage to the employee

A near miss is defined as any event that could have caused injury or damage to an employee, the public or property.

In the case of a minor injury, employees should be taken to Mercy medical center located at 6200 Whipple Ave. North Canton, Ohio, 44720 phone: 330-699-8884.

If the case of an employee with a major injury or illness immediately call a **“Code Blue”** over the PA system by dialing “88” announcing **“Code Blue”** and give the area where the employee can be located. Members of the first aid team will then respond and bring the Blood Borne Pathogen kit and the AED.

For all major injuries call 911 or the Jackson Township Fire Department at (330) 499-6666. When in doubt about the proper course of action, the accident should be treated as if it were major

First Aid Team

Several employees have been trained in basic First Aid treatments. Should these services be required, the first aid team is most easily contacted by dialing “88” and paging. When paging, the employee should give a detailed description of the location of the injured employee.

First Aid team members are listed on the Emergency Contact list found in Appendix A and posted in all departments and near the exits.

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3.4 Storms and Severe Weather

Severe thunderstorms, tornadoes or snowstorms are the most likely forms of severe weather encountered in northeastern Ohio.

A “watch” is less severe than a “warning.” As a rule of thumb, a “watch” means the type of severe weather has a high likelihood of occurring, while a warning means the storm has been sighted in the area.

3.4.1 Severe Thunderstorms and Tornadoes

In the event of a severe thunderstorm or tornado watch, the EHSC or designee will monitor the progress of the storm. Employees should be aware of the possibility of power outages and back up data or turn off instruments as appropriate.

If a tornado watch is upgraded to a warning, the EH&S or designee will be assigned to monitor the storm’s progress by radio and by watching the sky for visible funnel clouds. If a tornado is reported or seen within one mile of the facility, employees will be warned to go to their assigned assembly areas.

Employees should crouch on the floor, face to the floor, hands behind head and covered with any available soft items such as a coat. This position is known as the tornado crouch.

The EHSC will determine if it is safe to return to work. Employees are to remain in the assembly areas until the tornado has passed. In the event that there is building damage, employees will check for injuries, and if necessary evacuate the building.

3.4.2 Assembly areas.

Employees	Location
First floor offices GC VOA, MS VOA, FAS group	Hallway between cafeteria and receiving door, and GCV office.
Second floor and all non-VOA laboratory personnel	Main laboratory hallway between receiving and extractions.

4.0 Lockout / Tagout

Lockout / Tag out procedures establish the minimum requirements for the lockout of energy isolating devices whenever maintenance or servicing is done on machines or equipment. These procedures shall be used to ensure that the machine or equipment is stopped, isolated from all potential energy sources and “locked out” prior to an employee performing work on the equipment. These procedures are applicable for any servicing or maintenance where the unexpected energization or start-up of the machine or equipment or release of stored energy could cause the employee harm.

All employees are required to comply with the restrictions and limitations imposed upon them during the use of the lockout. Authorized employees are required to perform the lockout in accordance with this procedure. No employee, upon observing a machine or piece of equipment, which is locked out to perform servicing, shall attempt to start, energize, or use that machine or equipment. If any employee disregards this policy, he or she will be subject to TestAmerica disciplinary actions.

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The North Canton maintenance group has been trained in the proper use of Lockout Tagout.

Lockout / Tag Out Procedures

1. All affected employees must be notified that servicing or maintenance is required on a machine or piece of equipment and that the machine or equipment must be shut down and locked out to perform the servicing or maintenance. Notification will be in person, by phone or email and by posting an appropriate sign or notification
2. The Lab Manager, EH&S or Safety Coordinator, Maintenance Supervisor, or Operations Manager will be responsible for notifying group leaders in areas that will be affected by the lockout. The group leaders will then inform all personnel that could be potentially affected.
3. The authorized employee shall identify the type and magnitude of the energy that the machine or equipment utilizes, shall understand the hazards of the energy, and shall know the methods to control the energy. (Translation: They must know the equipment, how to disconnect it and what the consequences could be of it being turned on while they are servicing it)

There are several basic types of equipment requiring the Lockout Tagout Procedures

Electrical components such as:

- Facility lighting fixtures and switches.
- All emergency and exit lighting.
- All receptacles.
- Any blower, pump, etc. without a safety disconnect within 5 feet.

The following machines and or equipment are equipped with safety disconnect boxes mounted directly to the device and therefore do not require the Lockout / Tagout procedures

- All equipment on facility rooftop, including fume hood exhaust blowers, AC condensing units, cooling tower and ventilation fans.
- All fume hood makeup air supply blowers.
- All canopy hood suction blowers.
- All heat pumps located in facility plenum.
- All refrigeration condensing units located in building plenum.
- Facility makeup air supply units located in maintenance area.

General Procedures

If the machine or equipment is operating, shut it down by the normal procedure (depress the stop button, open switch, close valve, shut off breaker, etc.). If there is no safety disconnect box, the equipment should be de-energized at the breaker boxes.

Breaker box Locations

Utility chase
Total solids utility chase
Boiler room
Upstairs utility room.

1. Lock out the energy isolating devices, with assigned individual locks and tags.

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2. Stored or residual energy (such as that in capacitors, springs, elevated machine members, rotating flywheels, hydraulic systems, and air gas, steam, or water pressures, etc.) must be dissipated or restrained by methods such as grounding, repositioning, blocking, bleeding down, etc.
3. Ensure that the equipment is disconnected from all energy sources by first checking that no personnel are exposed, then verifying the isolation of the equipment by operating the normal controls and making certain that the equipment will not operate. Return controls to the neutral or off position. The equipment or machine is now locked out.
4. When the servicing or maintenance is completed and the machine or equipment is ready to return to normal operating condition, the following steps shall be taken.
5. The immediate area around the machine or equipment is checked to ensure that nonessential items have been removed and that the machine or equipment components are operationally intact.
6. Check the work area to ensure that all employees have been safely positioned or removed from the area.
7. Verify that the controls are in neutral or off position.
8. Remove the lock out devices and re-energize the equipment or machinery.
9. Notify the affected employees that the servicing or maintenance is completed and the machine or equipment is operational and ready for use.

5.0 Training

Safety training requirements for all employees are located in Appendix C of this document. A list of qualified trainers and qualifications can be found in this section.

6.0 Facility Transition and Exclusion Areas

6.1 Transition Areas

Transition areas are intermediate areas where employees wearing personal protective equipment (PPE) and employees not wearing PPE can pass. Any samples or chemicals passing through these areas must be in a closed container.

North Canton Transition Areas

- All hallways in the downstairs lab,
- The hallway between GC VOA and Corporate offices
- The hallway between the atrium and the downstairs rest rooms.
- Any area that is taped off with yellow and black striped tape.

6.2 Exclusion Areas

Exclusion areas are areas where employees must wear personal protective equipment. The entrance to these areas is taped off with yellow and black striped tape.

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7.0 Emergency Equipment Locations and Maintenance Requirements

7.1 Emergency Showers

Location

Emergency showers are located in every lab including the waste building (except GC VOA, and MS VOA). All showers are at least 100 feet, and / or 10 seconds from work areas. An emergency shower is also located in the waste building

Maintenance

Showers are checked weekly to ensure proper flow. The date of inspection is written on the card attached to the shower. Equipment for performing the check is kept in the warehouse.

7.2 Eyewashes

Location

Eyewashes are located in every lab under the emergency shower. Eyewashes in VOA's are located near the outside wall by the offices in GC/MS and at the sink near the hood in GC VOA. An eyewash is also located in the waste building

Maintenance

Eyewashes are checked weekly by running the water for a short time. The date of inspection is written on the tag attached to the shower or eyewash.

7.3 First Aid Kits

Location

North / south hallway of the main lab
1st east/ west hallway of main lab
Shower room
Nurse's office
Accounting area
Lunchroom

Maintenance

The nurse maintains First Aid kits.
An oxygen tank is located in the nurse's office.

7.4 Blood Borne Pathogen kits

Location

North / south hallway of the main lab
Lunch room
Nurse's office.

Maintenance

Blood Borne Pathogen kits are maintained by the nurse

7.5 Automatic External Defibrillator (AED)

Location

Outside the Nurse's office

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7.6 Spill Kits

Contents

Chemical suits

Gloves

Eye protection

Disposal bags

Absorbent booms and pads

Location

North / south hallway of the main lab (2)

Bullpen area, Small kits in each lab, Spill Cart outside of TCLP lab.

Waste Building Spill Equipment

Spill barriers for outside drains and absorbent pads

Chemical suits and gloves

Face and eye protection

7.7 Fire equipment

Location

- Emergency pull stations that call the fire department are located in all main hallways in the facility. These are marked and highly visible.
- Fire extinguishers are located in all main rooms of the facility and in hallways of smaller office rooms in the facility. They are marked and visible.
- All exits are marked with lighted Exit sign

Maintenance

- Fire extinguishers are checked monthly for intact seals. They are inspected and repaired by an outside company annually.
- The sprinkler system is checked quarterly by an outside company

7.8 Emergency ventilation

Emergency ventilation activate buttons are located at the ends of both east / west hallways in the main lab. A main activate button is located in the total solids utility chase located in front of the double doors before the main lab.

7.9 Hoods

Location

A table listing all North Canton hoods can be found in Appendix D

Maintenance

All hoods are checked semi-annually using a calibrated anemometer or velometer.

TestAmerica owned instruments are sent out to be calibrated according to the schedule recommended by the manufacturer.

7.0 Health and Safety Procedures for Handling Hazardous Wastes and Non Hazardous Waste

This document is to be used as the standard operating procedure (SOP) and training tool for hazardous waste procedures at TestAmerica North Canton.

Safety procedures shall be carried out in a manner that protects the health and safety of all TestAmerica North Canton employees, as well as the environment.

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PPE required for purging sample wastes

- Lab coat or apron
- Face shield
- gloves

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PPE required when bulking waste:

- Lab coat or apron.
- Face Shield
- Nitrile gloves with Butyl Gloves overtop.

Employees that process hazardous waste or operate glass crushers in the waste building will:

- Use the buddy system. No employee will attempt to process waste alone. Wear face shields, tyveks suits, booties and nitrile gloves with butyl gloves overtop.
- Be familiar with the locations of pull stations, fire extinguishers, showers, eyewashes, and spill response kits.
- Employees who handle broken glass from the glass crusher will wear “Blue Grip” gloves or a glove of a similar material.
- Ear muffers must be worn when operating the glass crusher.
 - ◆ The ear muffler must have a minimum rating of 26 decibels.
 - ◆ Failure to wear the hearing protection may result in loss of hearing and damage to the ear.
 - ◆ The employee must be trained on proper procedures for use of the hearing protection and must be given an annual hearing exam.

Special safety concerns when neutralizing waste:

- Face shields, tyveks suits, rubber booties, latex gloves with butyl gloves overtop.
- Slowly add Sodium Hydroxide to acid waste to prevent a viscous thermal reaction.

All of the above emergency equipment can be found in the drum storage building and the main facility. For spill response refer to Section 3 of the Facility Addendum. The drum storage building is equipped with the materials to handle a large spill.

A list of common solvents and their OSHA exposure limits and signs/symptoms of overexposure are listed in Appendix E.

Procedures for Receiving Waste from Satellite Accumulation Areas

Waste containers in the lab (satellites) will be checked and emptied on a weekly basis. The containers will be transported on a cart to the dock. From the dock the containers will be transported by cart to the drum storage building. Waste collected in 55-gallon drums will be carted from the sidewalk on the west side of the facility with a drum cart.

Before any lab waste is dispensed into the proper drum, the exhaust fans must be turned on in the drum storage building for employee protection. The lab waste from satellite containers will then be dispensed into the proper drum according to its contents. All metal drums will be grounded for spark protection when being filled.

After satellites are emptied, all drums will be capped and sealed. The building will then be securely locked so that no unauthorized personnel can access the building.

Proper Storage of Waste Material

TestAmerica North Canton is considered to be a large quantity generator by the EPA. No drum will be permitted to be in the drum storage building for over 90 days past the drum start date unless an exemption from the EPA is granted.

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The process for the proper storage of waste material is listed below:

- All drums and containers will be dated with a start date when transferred to the drum storage building.
- The drum will be labeled with the words **HAZARDOUS WASTE**.
- The label will document the contents of the drum along with the start date.
- The drum will be given a number and logged in the drum logbook.
- The drum number will be written in the corner of the hazardous waste accumulation label.
- The drums will be checked and accounted for weekly on a RCRA drum control sheet located in the logbook.
- All manifests and LDR's (land disposal restriction forms) will be signed by the facility Waste Coordinator, or an equally trained employee.
- Manifests, LDR's, annual EPA waste reports and RCRA drum control sheets will be retained for a minimum of 5 years.

Table 8.1

Waste Streams and Procedures for Preparation of Waste for Transportation to Disposal Facility	
Non detect solid samples. A.	Queried by receiving pulled after 90 days off the shelf in the cooler and put into DOT approved cubic yard waste container UN 11G / X / USA / +BR / 3306 / 1700 / 934. The samples are incinerated at an FDA approved incinerator. The cubic yard container is labeled with a non-regulated label. The proper shipping name is Non regulated, DOT non-regulated, none, none.
Non detect water samples. B.	Samples that are preserved are neutralized with sodium bicarbonate to a pH between 5 and 9 and drained to the local POTW. Neutral samples are drain to the local POTW.
<i>Volatile 40 ml water extracts</i> C.	Samples are crushed and separated from glass and plastic. The water is neutralized to a pH between 5 and 9 and sent to the local POTW.
Acidic extracts and waste. D.	The acid waste is drummed up in 55 gallon poly drums UN 1H1 / Y1.9 / 150 / USA / +1A . The drums are labeled with a class 8 corrosive label and a hazardous waste accumulation label, then neutralized with sodium hydroxide or sodium bicarbonate to a pH between 5 and 9 and drained to the local POTW.
Water samples with PCBs. E.	Receiving runs a query after the samples are analyzed. All samples above 50 ppm are purged into a 55 gallon steel drum UN 1A1/ X1.8 / 300 / USA /SDCC. The drum is labeled with a class 9 label, a "Caution contains PCBs" label and a hazardous waste accumulation label. The proper shipping name is Waste polychlorinated biphenyl's, liquid, 9, PCB2
Soil sample with PCB's. F.	Receiving runs a query after the samples are analyzed. All samples above 50 ppm are purged into a 55 gallon steel open top drum UN 1A2/ Y1.2/ 100 / USA.. The drum is labeled with a class 9 label, a "Caution contains PCBs" label and a hazardous waste accumulation label. The proper shipping name is Waste polychlorinated biphenyl's, solid, 9, PCB2
Solvent, oil, grease, multiphase and paint samples. G.	Drummed into a 55 gallon steel open top drum UN 1A2 / Y1.2 / 100 / USA. The drum is labeled with a class 3 flammable liquid label and a hazardous waste accumulation label The proper shipping name is Waste flammable liquids, corrosive, N.O.S.

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	(paint thinner nitric acid), 3, UN2924, I, D001, D002.
Unusual sample waste from results of analysis. H.	Samples that meet characteristics of 40 CFR 261 subpart C and D, are collected for a lab pack and properly disposed of. Example, benzene's, cyanides, mercuries, etc.
Flammable liquid waste I.	Acetone, Acetonitrile, Methanol, Ether, and hexane wastes combined into a 55 gallon steel drum UN 1A1 / X1.8 / 300 / USA / SDCC. The drum is label with a class 3 flammable liquid label and a Hazardous waste accumulation label. The proper shipping name is Flammable Liquid N.O.S., (ether, acetonitrile), 3, UN1993, I, D001 F003.

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Table 8.1 (Continued)

Waste Streams and Procedures for Preparation of Waste for Transportation to Disposal Facility	
Dichloromethane waste from extracting water samples J.	The water samples with the left over dichloromethane waste is poured into an EPA approved liquid/liquid separation unit, which was developed at the North Canton lab. When the liquid/liquid separation unit is full, it is taken to the tank room where the water is neutralized to a pH between 5 and 9. The unit connects to a drain system that leads to a 55 gallon steel drum UN 1A1 / X1.8 / 300 / USA / SDCC. in a storage unit outside of the building to prevent exposure. Using a valve system on the separation unit then visually drains off the dichloromethane layer. The neutralized water layer is then drained to the local POTW. The drum is labeled Class 6.1 poison label and a hazardous waste accumulation label. The proper shipping name is Waste Dichloromethane, 6.1, UN1593, III, F002.
Mixed dichloromethane and flammable liquids K.	The waste contains dichloromethane, acetone, and hexane. The waste is collected in a 25 gallon DOT approved container and taken out to the drum storage building on a weekly basis. The waste is then transferred to a 55 gallon steel drum UN1A1 / X1.8 / 300 / USA / SDCC. The drum is labeled with a Class 3 flammable liquid label, a class 6.1 poison label and hazardous accumulation label. The proper shipping name is Waste Flammable liquids, toxic N.O.S., (Acetone, methylene chloride), 3, UN1992, II, D001, F002, F003.
Sulfuric acid waste with less than 1 % hexane from PCB clean up L.	The sulfuric acid is separated off from the sample extract. The waste is poured into a 5 gallon poly UN 1H1 / Y1.8 / 100 / USA /. When the container is full, it is transferred to the drum storage building. The container is labeled with a class 8 corrosive label and a hazardous waste accumulation label. The proper shipping name is Waste corrosive liquids, N.O.S. (Sulfuric acid, hexane), 8, UN2920, II, D002.
C.O.D. waste M.	The waste is collected into a 5 gallon poly UN 1H1 / Y1.8 / 100 / USA /, and then transferred out to the waste building. The container is labeled with a class 8 corrosive label and a hazardous waste accumulation label. The proper shipping name is Waste corrosive liquids, toxic, N.O.S. (sulfuric acid, mercuric sulfate) 8, UN2922, D002, D007, D009, D011.
Sample extract vial waste N.	The sample extracts contain dichloromethane, hexane, acetone and acetonitrile. The 2 to 12 milliliter vials are put directly into a 55 gallon steel open top drum UN 1A2 / Y1.2 / 100 / USA. . The drum is transported directly to the drum storage building. The drum is labeled with a class 3 flammable liquid label and a hazardous waste accumulation label. The proper shipping name is Waste flammable liquids N.O.S., (hexane, acetone), 3, UN1993, II, D001, F002, F003.

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Table 8.1 (Continued)

Waste Streams and Procedures for Preparation of Waste for Transportation to Disposal Facility	
Sample extract vial waste containing PCB's O.	The sample extracts contain dichloromethane, hexane, acetone and acetonitrile. The 2 to 12 milliliter vials are put directly into a 55 gallon steel drum UN 1A2 / Y1.2 / 100 / USA . The drum is transported directly to the drum storage building. The drum is labeled with a class 3 flammable liquid label and a hazardous waste accumulation label. The proper shipping name is Waste flammable liquids N.O.S., (hexane, acetone), 3, UN1993, II, D001, F002, F003, PCB2.
Mercury waste P.	The mercury waste is collected into a 1 liter container. It is then transferred into a 5 gallon poly container UN1H1 / Y1.8 / 100 / USA. The container is labeled class 8 corrosive label and a hazardous waste accumulation label. The proper shipping name is Waste mercury, 8, UN2809, III, D009.
Pyridine and water waste Q.	The waste is collected into a 25 gallon poly container UN 1H1 / Y 1.9 / 150 / USA/ +A1. It is directly transported to the Drum storage facility. The drum is labeled with a class 9 label and a hazardous waste accumulation label. The proper shipping name is Hazardous waste liquid, N.O.S. (pyridine cyanide), 9, NA3082, III, D002, D038, F005.
Lab Debris R.	The waste consists of weigh tins, gloves, thimbles, extracted soil, rags and paper towels. The waste is non-regulated and shipped to a FDA approved incinerator. The waste is collected in 20 gallon solid debris waste containers. It is then transferred into a cubic yard container UN 11G / X / USA / +BR / 3306 / 1700 / 934. The container is labeled with a Non-regulated waste label. The proper shipping name is Non regulated, DOT Non regulated, none, none
Expired reagents, standards, and chemicals X.	These items are segregated by storage compatibility, and dated to be lab packed and sent out for proper disposal from an outside source.
All waste will be drummed into D.O.T. approved containers and drums for disposal.	

Description of 90 day storage area.

The 90 day waste accumulation area is the white brick building in the south east parking lot from the facility. The facility is equipped with a spill kit, fire extinguisher, eye wash and safety shower. The ventilation fans, lights and heater are explosion proof. The metal grated floor is grounded.

Corrosives will be stored on the east part of the building. Solvents will be stored toward the west side of the building. Non compatibles with corrosive or solvent material will be stored on the north side of the building.

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Satellite Accumulation areas

WASTE STREAM GENERATED AT TESTAMERICA NORTH CANTON			
WASTE ID	WASTE DESCRIPTION	SSA CONTAINER	MAIN COLLECTION PROCESS OR CONTAINER
I	Acetone, Acetonitrile, Methanol, Ether and Hexane.	20-Liter poly carboys	55-Gallon Steel Drum
R	Lab Debris	20-Gallon Waste Container	Cubic Yard
G	Solvent, Oil, Grease, Multiphase and Paint Samples.	20-Liter poly Carboys	55 –Gallon Steel Drum (Open Top)
J	Dichloromethane	25 Gallon Waste Container	55-Gallon Steel Drum
P	Mercury Waste	1 Liter	5 Gallon Poly Container
L	Sulfuric Acid	5 Gallon Poly	Same Container
O	Sample Extract	55-Gallon Steel Drum	Same Drum
D	Acidic Extracts	55-Gallon Poly Drum	Same Drum
M	COD Waste	5 Gallon Poly	Same Container
Q	Pyridine and Waste Water	25 Gallon Poly	Same Container
K	Mixed solvents.	25 Gallon Waste Container	55-Gallon Steel Drum

SATELLITE ACCUMULATION AREAS (SSAs) AND WASTE GENERATED AT THE SAA	
SAA	WASTE STREAM GENERATED
MS VOA	I, R
GC VOA	G, I, I, R
GC (Instrument Room)	I
GC Semi (Prep Room)	P, L, I, O
WC (Instrument Room)	Q, M, D
WC (BOD Room)	O
WC (Prep Room)	I,
MSS	O
Extractions	I, J, K

Personal authorized to handle hazardous waste: Steve Jackson, Eric Mills

8.2 Control of possible or Known Hazardous samples.

Possible or known hazardous samples will be evaluated on project and or hazard basis by EH&S, the technical director, the project manager, and management when needed. The samples will remain in strict control to ensure employee safety.

Once necessary evaluations are done for proper log in (i.e. Unpacking or initial diluting in an engineered environment) the sample will be stored in the caged area under lock and key.

Only authorized personnel will have control and access to the cage. These people will be the Sample Management supervisor, Organic Extractions manager, Metals manager, Wet Chemistry

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manager, Voa's Managers and EH&S. A high level chemist may be put in charge of control of the samples under their managers supervision.

All controlled samples will be returned to the cage area by the employee that took them out.

8.3 Destruction of Known hazardous samples or samples for litigation.

If requested by the client or project manager special projects such as samples for litigation can be tracked. Otherwise all samples will be considered disposed of per cradle to grave per the normal disposal procedures in place.

The procedure for individual sample tracking will be each sample container being flagged with a marking or label. When sample retention is up or the project has been given the permission to be disposed of , sample management will record each sample container and record the drum number the container went into.

This document will be kept in the control of the waste coordinator to be stored with the Hazardous Waste Manifests with the shipment the samples went out with. The drum number is also tracked electronically in the waste database system which shows the drum number, the date, and the manifest the drum was destroyed.

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9.0 Working Alone

9.1 Laboratory Personnel

No one is permitted to work alone in any non-administrative portion of the lab unless the procedure described below is followed. It is recommended that people working alone in administrative areas also follow this procedure.

A person is considered working alone when: they are the only person in the building or when they are the only person working in an area that is not within voice range of other persons in the building.

Procedure

This procedure shall apply to all employees working in a non-administrative area of the building on off-shifts, weekends or holidays.

1. Each person entering the building shall sign-in using the log located at the side door entrance.
2. The first person entering the building shall pick-up the walkie-talkie located in the utility chase area outside of the sample receiving office. The walkie-talkie will be carried at all times.
3. The first person to sign-in for each area of the building shall pick-up a walkie-talkie and turn it on to channel #2. The person will carry the walkie-talkie at all times. Note: Each area will be assigned a walkie-talkie. The walkie-talkie and its charger will be identified with the area's name.
4. If an emergency arises:
 - a. Notify others in the lab using the walkie talkie or paging system if outside assistance is needed, and a telephone can be reached, request emergency help by dialing 9-911.
5. Whenever a person in possession of a walkie-talkie leaves the building, the walkie talkie will be handed over to another person working in the same area. The last person to leave the area for the day shall return the walkie talkie to its charger.
6. Each person exiting the building shall sign out on the log sheet.

9.2 FAS Personnel

FAS personnel and couriers shall:

- Be required to carry a cellular telephone while performing work outside of the TestAmerica-North Canton laboratory.
- Understand the client's emergency reporting and evacuation procedures.
- Follow client sign-in/sign-out procedures
- If not returning to lab at the end of the day, contact supervisor when reaching home.


10.0 Documentation Matrix


A matrix listing the various EHS documents and their file location is included in Appendix F.

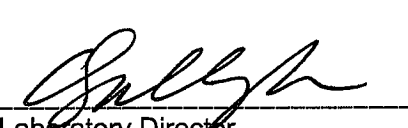
Title: PROJECT MANAGEMENT

[Method: None]

Approvals (Signature/Date):

 8/1/08
Technology Specialist Date

 08/01/08
Quality Assurance Manager Date

 8/7/08
Laboratory Director Date

This SOP was previously identified as SOP No. NC-AD-0001, Rev 1.2, dated 07/23/07

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1. PURPOSE

- 1.1. To describe the management of projects from the acceptance of the initial client request through the issuance of the final report and invoice.
- 1.2. This document accurately reflects current Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. RESPONSIBILITIES

- 2.1. It is the responsibility of the employee to perform the procedure described herein in full compliance with this SOP.
- 2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of this facility to assure that the tasks described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.

3. SAFETY

- 3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

4. PROCEDURES

- 4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.
- 4.2. Standard office equipment and supplies are used in the day to day tasks of a Project Manager (PM). This would include a designated PC with internet connection and telephone, along with access to a scanner, fax machine, and copier.
- 4.3. QuantIMS quote generation
 - 4.3.1. All projects are set up in TestAmerica North Canton QuantIMS database using the customer service quote maintenance module. Creation of the QuantIMS quote is the foundation of all project setup and initiation activities by the PM. Information in the quote includes the structured analysis codes for the specific analyses, along with pricing, naming conventions, compound lists, report and invoice routing information, report formatting and data deliverables options along with any special instructions. Quotes are used by the PM to establish all of the specific parameters around a given project. They will serve as a confirmation, when faxed

or emailed to the client, of our capability to meet their project requirements as well as a formal written cost proposal.

- 4.3.2. To ensure all aspects of the project are correct, a Project Setup Checklist (WI-NC-017A-070907) is completed for all new projects.
- 4.3.3. For a complete description of how to use the customer service quote module, reference the QuantIMS Quote Module Training Manual.
- 4.4. Set up and delivery of sampling supplies
 - 4.4.1. In most cases, TestAmerica North Canton provides clients with the appropriate sample containers, preservatives, documentation, and packing material for return shipment for all samples to be analyzed by the laboratory. TestAmerica North Canton Shipping group performs the packaging and shipping of the appropriate supplies. They complete their duties in accordance with SOP NC-QA-012, Shipping Department SOP. It is the PM's responsibility to provide the shipping group with a written request in a format suitable for processing. The required supply request form is to be completed electronically by the PM and emailed to the shipping group. A copy of that form is included as Appendix 6.2 to this SOP. In addition to the shipping address, due date, and specific request information, all supply request forms must contain the QuantIMS quote number for that project.
- 4.5. Resolution of Sample Receiving Issues
 - 4.5.1. When samples arrive at the laboratory, TestAmerica North Canton Sample Receiving group processes them from arrival through sample log-in and to their final disposition in sample storage while waiting to be analyzed. Sample log-in will be initiated when the PM supplies the Receiving group with the correct quote information. All activities of the sample receiving group are performed in accordance with Sample Receiving SOP NC-SC-005. Included in their procedure is the identification of anomalies, nonconformances, and questions. In each case, the Receiving group either communicates the issue and their corrective action to the PM for approval, or they seek resolution of an issue by the PM. It is the PM's responsibility to approve or resolve all issues brought to their attention by the Receiving group.
 - 4.5.2. In cases where the PM needs to provide specific information to the Receiving group or resolve problems related to the condition of samples, the Receiving group will set aside the samples related to the project in question until the PM provides them with the needed direction. It is the PM's responsibility to work with clients to resolve any outstanding issues then to direct the Receiving group toward the appropriate course of action.

- 4.6. Approval of the sample log in process and preliminary invoice
 - 4.6.1. After the sample receipt and log-in process is complete, a summary report is generated from the data base that displays the information entered during log-in. This report is attached to all of the documentation that accompanies the samples to the lab along with the Cooler Receipt form, preliminary invoice, and a project checklist generated by the Receiving group. The log-in is reviewed for accuracy by the Receiving group, and the client analysis summary is then forwarded to the person designated to scan copies of the Chain-of-Custody and Cooler Receipt forms into the electronic project archive. Documents are scanned within one business day, and the summary is relinquished through the intranet summary tracking system and forwarded to the appropriate PM. It is the responsibility of the PM to review this summary report along with the preliminary invoice within a day of receiving it. If any errors are found, it is the PM's responsibility to correct or communicate the error to the Receiving group for immediate correction. The PM approves the summary by initialing the front page, relinquishes the summary through the intranet summary tracking system, and forwards it on to the Reporting group. The PM approves the preliminary invoice by initialing it as well.
 - 4.6.2. The project checklist must be reviewed for completeness by the PM. All of the relevant routing information must be contained. This includes the format and number of report copies, type of EDDs if applicable, invoice routing information, and whether a paginated report and CD are required. A copy of the project checklist is included as Appendix 6.3 to this SOP.
- 4.7. Tracking and maintaining delivery schedules
 - 4.7.1. It is the responsibility of the PM to monitor project schedules and communicate with operations to obtain data that is at risk of being late. In cases where the PM knows that delivery schedules will not be met by operations, they are to contact the client and provide them with a revised delivery schedule. After notifying the client, the PM will change the due date in the system, if appropriate. The PMs track their delivery schedules in QuantIMS using a management report (MGT: M36) they run daily from the AS400 database. This report lists every project the PM has in-house along with the analytical due dates, report due dates, and number of days left before final reports must be mailed.
 - 4.7.2. A Rush Notification Form is used for all rush work. A copy of the form is located in Appendix 6.4. Details of the procedure are documented in Work Instruction WI-NC-002/A-10/03/05
- 4.8. Final review and approval of the final report and invoice
 - 4.8.1. When final reports are issued by the Reporting group, the PM must review the

case narrative against the various check lists, NCMs, and receiving documentation held with the project file, to verify its completeness and accuracy. If additions or corrections are required, the PM is to correct or communicate the error to the Reporting group for immediate correction. The PM must also verify that the draft invoice contained in the project file is complete and accurate. When the review is complete, the PM is to sign the report cover at the front of the package.

- 4.8.2. Upon receiving report packages from the reporting group, it is the responsibility of the PM to limit the dwell time of that report package to less than 1 day before final approval, invoicing and shipment to the client.
- 4.8.3. After completing the final approval of the report and invoice, the PM gives the entire package to the Project Assistants (PA). The PAs manage the completion of the copying, scanning, invoicing and shipping tasks for all final reports and invoices.

5. DEFINITIONS

- 5.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version

6. APPENDICES

6.1. References

- 6.1.1. Sample Receiving and Sample Control SOP NC-SC-005, current version
- 6.1.2. Shipping Department SOP NC-QA-012, current version
- 6.1.3. Supplemental Practices for DoD Project Work, NC-QA-0016, current version
- 6.1.4. TestAmerica North Canton Quality Assurance Manual (QAM), current version
- 6.1.5. TestAmerica Corporate Safety Manual, M-E-0001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version
- 6.1.6. Project Setup Checklist, Work Instruction WI-NC-017A-070907
- 6.1.7. Rush Request, Work Instruction WI-NC-002/A-10/03/05
- 6.1.8. Revision History

Historical File:

Revision 1.0: 01/21/04

Revision 1.2: 07/23/07

Revision 1.1: 03/02/05

Revision 1.3:

6.2. Example: Supply Request Form

SUPPLY REQUEST FORM

QUOTE #:

SITE:

BILLING ADDRESS

SHIPPING ADDRESS

Client Code:

Client Name:

Address:

Telephone:

Contact:

Client to be Charged For:

☐ Supplies

☐ Shipping

☐ No Charge

Supply Charges:

Shipping Charges:

Date Charged:

Lot #:

PO#:

TestAmerica Shipper Requested:

☐ Cooler

☐ Sample Shuttle

☐ Box

Date Needed: a.m.

Number of Shippers:

Shipment Method:

Weight:

Description of Shipment:

Name:

Date:

6.3 Example: Project Manager Report Checklist**LEVEL III REVIEW - PROJECT MANAGEMENT CHECKLIST**

Rev 06

04/19/02

INVOICE TO: _____ PHONE: _____

Client Name _____ Invoice with Report ☐ Invoice Separate ☐ Hold Invoice ☐

SITE NAME: _____

PROJECT NUMBER: _____

Client Code: _____ EDD Needed: Yes ☐ No ☐ CD ROM ☐Diskette Needed: Yes ☐ No ☐

Lot # _____ EDD Instructions _____

PM _____ Other _____

Report Due Date _____ Original C-O-C with Report Yes ☐ No ☐Paginate ☐ # of Copies EXP _____ Unbound ☐ Stapled ☐Paginate ☐ # of Copies STD/SUM _____ Unbound ☐ Stapled ☐Scan and Email final report only Yes ☐ No ☐

Reports to: _____

Comments: _____

- | | |
|--|---|
| <input type="checkbox"/> Sample IDs on cover | <input type="checkbox"/> Number of waters |
| <input type="checkbox"/> TIC pages included | <input type="checkbox"/> Number of soils |
| <input type="checkbox"/> Results need dry weight basis | <input type="checkbox"/> Number of wastes |
| <input type="checkbox"/> Executive Summary | <input type="checkbox"/> Number of trip blanks |
| <input type="checkbox"/> Method Analyst Summary | Out of lab data included Yes <input type="checkbox"/> No <input type="checkbox"/> |
| <input type="checkbox"/> Field Report | |

Narrative Review By: _____ Date _____

PM has reviewed this report for the above criteria _____ Date _____

KRI INFORMATION

1. Was report mailed on time? YES or NO

If no, why? _____

2. Was fax required? YES or NO Was it faxed on time? YES or NO

If no, why? _____

6.4. Example: Rush Notification Form (WI-NC-002A-100305)

Status: *Original*

Date: Pre-approved by Ops Yes

Project Manager: Calovini

Project Assistant: Brooks

Client:

Site:

Quote#:

TAT: 1 Day

Estimated Arrival Date:

Due Date:

Multiple Delivery Dates: No

If Yes, Over How Many Days: Not Applicable

TOTAL Number of samples:

Number of Water samples:

Number of Solid samples:

Number of Wipe samples:

Tests, select (click☺) all that apply:

❖ MS Volatiles: ☐ 8260 ☐ 624 ☐ CLP ☐ Other (list below)

❖ MS Semivolatiles: ☐ 8270 ☐ 625 ☐ SIM ☐ Other (list below)

❖ GC Volatiles: ☐ 8021 ☐ 601/602 ☐ TPH/GRO ☐ WI/GRO ☐ Other (list below)

❖ GC Semi: ☐ 8082 ☐ 8081 ☐ 8310 ☐ 8151 ☐ 8141 ☐ TPH/DRO ☐ WI/DRO ☐ Other

❖ Metals: ☐ ICPMS ☐ ICP ☐ Mercury ☐ Low-Level Mercury ☐ Other (list below)

- ❖ General Chemistry: ☐ Cyanide ☐ IC ☐ TOX ☐ HEM ☐ TS ☐ Other (list below)
- ❖ ☐ TCLP ☐ SPLP(☐ East/☐ West) ☐ 8270 ☐ 8081 ☐ 8151 ☐ Metals ☐ Other (list below)
- ❖ ZHE: ☐ 8260
- ❖ Reporting: ☐ Prelims ☐ Locked results ☐ Rush Data Package TAT: N/A

.....

Comments:

6.5. Example: PROJECT SETUP CHECKLIST (WI-NC-017A-070907)

Name of Project: _____ Quote #:

Type of Project: ☐ RCRA
☐ Ohio VAP
☐ NPDES
☐ DOD QSM ☐ LCG
☐ Clean Water Act
☐ Ohio Landfill requirements: PQLs, PQL blurb, QDS report,...
☐ Other _____

Is there a QAPP for this project? Yes ☐ or No ☐

Certifications required? Yes ☐ or No ☐ If so, which ones?

Frequency: ☐ One-time Event
☐ Quarterly
☐ Semi-Annual
☐ Annual
☐ Other _____

Estimated Start Date:

“Bill to” address

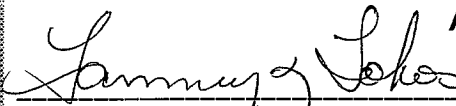
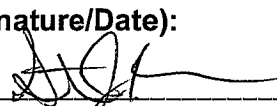


“Report to” address

Required Turn Around Time: ☐ Standard (21 days report)

- Company Confidential & Proprietary**

Title: CENTRAL REPORTING

[Method: None]

Approvals (Signature/Date):			
	7/14/08		7/16/08
Technology Specialist	Date	Health & Safety Coordinator	Date
	7/16/08		7/15/08
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-AD-002, Rev 1, dated 04/24/02

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Facility Distribution No. _____

Distributed To: _____

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1. PURPOSE

- 1.1. To describe the assembly of a report package including the completion of analytical data, compilation of data from each lab group, final completeness review, and preparation for invoicing and mailing. Specific client requirements are incorporated in each step of the process.
- 1.2. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. RESPONSIBILITIES

- 2.1. It is the responsibility of the employee to perform the procedure described herein in full compliance with this SOP.
- 2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of this facility to assure that the analysis described is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.

3. SAFETY

- 3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

4. PROCEDURES

- 4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.
- 4.2. Folders
 - 4.2.1. The Records Manager creates labels and folders for each group.

The Records Manager prints a set of labels each day. A label is generated for each project lot that was logged into LIMS the previous day. The labels are given to a designated staff member to put on the blank folders. When complete, the folders are given to Central Reporting and query sheets are stapled on the outside of the folder. The folders are filed numerically.

4.3. Expanded Deliverables - Metals Procedure

4.3.1. When all the metals data requested for a specific lot or SDG has been analyzed, completed, and reviewed/released, the metals data package can be compiled. All metals raw data which includes analytical runs, batch sheets, and prep logs are either scanned or electronically pdf'd to the Reporting drive. Reporting runs a query which identifies which analytical runs and raw data is needed for a lot or SDG. Bubbaware software and LIMS are used to create QC Forms (Nos. 1-14). These forms are checked for accuracy and printed to pdf. A program is run which pulls the raw data, Bubbaware forms, and LIMS forms while generating the Chain-of-Custody so the package can be composed electronically. The program also pulls the Bubbaware forms and LIMS forms to generate a summary package, which is posted to the Project drive. The expanded package is checked for completeness and posted to the Project drive. It is then logged as "done" for metals package.

4.4. Expanded Deliverables -Wet Chemistry Procedure

- 4.4.1. When all of the samples in a lot (or SDG) are reviewed/released, the package for the expanded deliverable can be assembled.
- 4.4.2. All Wet Chemistry raw data is scanned or pdf'd electronically by batch.
- 4.4.3. A program is run that identifies which batches were reported for the lot or SDG. It copies the raw data of the batches to the Reporting drive. The technician generates a pdf of Wet Chem results from LIMS.
- 4.4.4. A review is done on the report. Holding times, parameter relationship issues, batch QC, dilutions, flags and non-conformance memos are checked.
- 4.4.5. Another program is run that pulls all Wet Chem results, raw data associated and generates Chains-of-Custody, and composes it into a single pdf document. A summary package, which includes LIMS results, is also posted to the Project drive by the program. The package is checked for completeness, and posted to the Project drive. It is then logged a "done" for Wet Chemistry.

4.5. Expanded Deliverables - Organics Procedure

- 4.5.1. The lab generates a pdf electronically for all acceptable raw data run on their instruments and processed under the Target system. All sequence logs are scanned to individual lab group folders.

- 4.5.2. When a lot or SDG is completed by the lab, it is identified on an automatic spreadsheet in Reporting.
- 4.5.3. Reporting runs a program identifying when samples were run for a method, including dates. The technician uses the report and Target to decide which Initial calibrations are required in the package. They copy the Initial calibrations, Continuing calibrations, Tuning calibrations, and sequence logs to a specific folder on the Reporting drive.
- 4.5.4. The technician prints the LIMS report of the final data to pdf, which represents Form 1's. They are checked for completeness and invalid data lines. They also print Forms 2-4 of the method, and scan them to pdf. Forms 2-3 are labeled as QC, and put into the folder on the Reporting drive.
- 4.5.5. Forms, 5, 8, and 10 are generated using Target Envisions. They are printed and scanned to the drive.
- 4.5.6. A program is run that pulls the Target pdf of raw sample data, LIMS reports, and pdf's, which are in a specific folder (Forms 2-4, Target Forms, Initial Calibrations, Continuing Calibration, Tuning Calibrations, and sequence logs), and composes them into a single pdf. The program also generates the bench worksheet and Chain-of-Custody, which is included in the single pdf with other associated data.
- 4.5.7. The program generates a summary package, which consists of Form 1's, and places it in the Project drive.
- 4.5.8. The pdf is checked for completeness, and moved to the Project drive.
- 4.6. Final Prep Prior to Narrative
 - 4.6.1. The last group finishing the package for that lot or SDG runs the final report checker, which checks for Non-Conformance Memos (NCMs) and any added parameters. It is logged into narrative tracking and moved to the Narrative shelf.
- 4.7. For Non-Expanded Reports
 - 4.7.1. A query is run at least once a day that lists all the lots that are reviewed/released, but are not invoiced. Using the previous day's query, the lists are compared, and all new lots appearing are marked. The project folders for these lots are pulled from the shelf. The result pages are printed to pdf for all the lots pulled. The standard report program is run for all lots. It prints out a checker sheet, which

lists all batches associated with the sample, any NCMs, and Wet Chem holding times.

- 4.7.2. The technician checks the pdf for completeness, for invalid data lines, for Wet Chem holding times, and for sufficient QC for the batches. When everything meets requirements, the technician moves the report to the Project drive. It is logged into narrative tracking, and moved to the Narrative shelf.

4.8. Narratives

- 4.8.1. The information contained in the narrative is derived from all the documentation received from the client, sample log-in, and data received from the individual laboratories.
- 4.8.2. The Narrative Generator program is run for the lot or SDG. It pulls in all client information and any flagging that was done on LIMS. The technician adds any NCM information and any narrative information received from the lab. The cooler temps and any information from the Cooler Receipt Form is added. The narrative is saved and printed to pdf.
- 4.8.3. An updated cover is generated, and the narrative is printed for Project Manager review.
- 4.8.4. An Electronic Deliverable is processed if required. The completed report is then presented to the Project Manager for review.

5. DEFINITIONS

- 5.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version

6. APPENDICES

- 6.1. Appendix 1 – Quality Control Elements Narrative
- 6.2. Appendix 2 – Example of Expanded Deliverable Tracking Sheet
- 6.3. Appendix 3 – Example of Narrative Tracking Checklist
- 6.4. Appendix 4 – Wet Chemistry Parameter Relationship Checklist
- 6.5. References

6.5.1. TestAmerica North Canton Quality Assurance Manual (QAM), current version

6.5.2. Revision History

<u>Historical File:</u>	Revision 0: 08/17/01	Revision 3:
	Revision 1: 04/24/02	Revision 4:
	Revision 2:	

APPENDIX 1

QUALITY CONTROL ELEMENTS NARRATIVE

QUALITY CONTROL ELEMENTS NARRATIVE

TestAmerica North Canton conducts a quality assurance/quality control (QA/QC) program designed to provide scientifically valid and legally defensible data. Toward this end, several types of quality control indicators are incorporated into the QA/QC program, which is described in detail in QA Policy, QA-003. These indicators are introduced into the sample testing process to provide a mechanism for the assessment of the analytical data.

QC BATCH

Environmental samples are taken through the testing process in groups called QUALITY CONTROL BATCHES (QC batches). A QC batch contains up to twenty environmental samples of a similar matrix (water, soil) that are processed using the same reagents and standards. TestAmerica North Canton requires that each environmental sample be associated with a QC batch.

Several quality control samples are included in each QC batch and are processed identically to the twenty environmental samples.

For SW846/RCRA methods, QC samples include a METHOD BLANK (MB), a LABORATORY CONTROL SAMPLE (LCS) and, where appropriate, a MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) pair or a MATRIX SPIKE/SAMPLE DUPLICATE (MS/DU) pair. If there is insufficient sample to perform an MS/MSD or an MS/DU, then a LABORATORY CONTROL SAMPLE DUPLICATE (LCSD) is included in the QC batch.

For 600 series/CWA methods, QC samples include a METHOD BLANK (MB), a LABORATORY CONTROL SAMPLE (LCS) and, where appropriate, a MATRIX SPIKE (MS). An MS is prepared and analyzed at a 10% frequency for GC Methods and at a 5% frequency for GC/MS methods.

LABORATORY CONTROL SAMPLE

The Laboratory Control Sample is a QC sample that is created by adding known concentrations of a full or partial set of target analytes to a matrix similar to that of the environmental samples in the QC batch. Multi peak responders may not be included in the target spike list due to co-elution. The LCS analyte recovery results are used to monitor the analytical process and provide evidence that the laboratory is performing the method within acceptable guidelines. All control analytes indicated by a bold type in the LCS must meet acceptance criteria. Failure to meet the established recovery guidelines requires the reparation and reanalysis of all samples in the QC batch. Comparison of only the failed parameters from the first batch are evaluated. The only exception to the rework requirement is that if the LCS recoveries are biased high and the associated sample is ND (non-detected) for the parameter(s) of interest, the batch is acceptable.

At times, a Laboratory Control Sample Duplicate (LCSD) is also included in the QC batch. An LCSD is a QC sample that is created and handled identically to the LCS. Analyte recovery data from the LCSD is assessed in the same way as that of the LCS. The LCSD recoveries, together with the LCS recoveries, are used to determine the reproducibility (precision) of the analytical system. Precision data are expressed as relative percent differences (RPDs). If the RPD fails for an LCS/LCSD and yet the recoveries are within acceptance criteria, the batch is still acceptable.

METHOD BLANK

The Method Blank is a QC sample consisting of all the reagents used in analyzing the environmental samples contained in the QC batch. Method Blank results are used to determine if interference or contamination in the analytical system could lead to the reporting of false positive data or elevated analyte concentrations. All target analytes must be below the reporting limits (RL) or the associated sample(s) must be ND except under the following circumstances:

- Common organic contaminants may be present at concentrations up to five times the reporting limits. Common metals contaminants may be present at concentrations up to two times the reporting limit, or the reported blank concentration

must be twenty-fold less than the concentration reported in the associated environmental samples. (See common laboratory contaminants listed in the table.)

Volatile (GC or GC/MS)	Semivolatile (GC/MS)	Metals ICP-MS	Metals ICP Trace
Methylene Chloride, Acetone, 2-Butanone	Phthalate Esters	Copper, Iron, Zinc, Lead, Calcium, Magnesium, Potassium, Sodium, Barium, Chromium, Manganese	Copper, Iron, Zinc, Lead

- Organic blanks will be accepted if compounds detected in the blank are present in the associated samples at levels 10 times the blank level. Inorganic blanks will be accepted if elements detected in the blank are present in the associated samples at 20 times the blank level.
- Blanks will be accepted if the compounds/elements detected are not present in any of the associated environmental samples.

Failure to meet these Method Blank criteria requires the re-preparation and reanalysis of all samples in the QC batch.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE

A Matrix Spike and a Matrix Spike Duplicate are a pair of environmental samples to which known concentrations of a full or partial set of target analytes are added. The MS/MSD results are determined in the same manner as the results of the environmental sample used to prepare the MS/MSD. The analyte recoveries and the relative percent differences (RPDs) of the recoveries are calculated and used to evaluate the effect of the sample matrix on the analytical results. Due to the potential variability of the matrix of each sample, the MS/MSD results may not have an immediate bearing on any samples except the one spiked; therefore, the associated batch MS/MSD may not reflect the same compounds as the samples contained in the analytical report. When these MS/MSD results fail to meet acceptance criteria, the data is evaluated. If the LCS is within acceptance criteria, the batch is considered acceptable.

For certain methods, a Matrix Spike/Sample Duplicate (MS/DU) may be included in the QC batch in place of the MS/MSD. For the parameters (i.e. pH, ignitability) where it is not possible to prepare a spiked sample, a Sample Duplicate may be included in the QC batch. However, a Sample Duplicate is less likely to provide usable precision statistics depending on the likelihood of finding concentrations below the standard reporting limit. When the Sample Duplicate result fails to meet acceptance criteria, the data is evaluated.

For certain methods (600 series methods/CWA), a Matrix Spike is required in place of a Matrix Spike/Matrix Spike Duplicate (MS/MSD) or Matrix Spike/Sample Duplicate (MS/DU).

The acceptance criteria do not apply to samples that are diluted.

SURROGATE COMPOUNDS

In addition to these batch-related QC indicators, each organic environmental and QC sample is spiked with surrogate compounds. Surrogates are organic chemicals that behave similarly to the analytes of interest and that are rarely present in the environment. Surrogate recoveries are used to monitor the individual performance of a sample in the analytical system.

If surrogate recoveries are biased high in the LCS, LCSD, or the Method Blank, and the associated sample(s) are ND, the batch is acceptable. Otherwise, if the LCS, LCSD, or Method Blank surrogate(s) fail to meet recovery criteria, the entire sample batch is reprepared and reanalyzed. If the surrogate recoveries are outside criteria for environmental samples, the samples will be reprepared and reanalyzed unless there is objective evidence of matrix interference or if the sample dilution is greater than the threshold outlined in the associated method SOP.

The acceptance criteria do not apply to samples that are diluted. All other surrogate recoveries will be reported.

For the GC/MS BNA methods, the surrogate criterion is that two of the three surrogates for each fraction must meet acceptance criteria. The third surrogate must have a recovery of ten percent or greater.

For the Pesticide and PCB methods, the surrogate criterion is that one of two surrogate compounds must meet acceptance criteria. The second surrogate must have a recovery of 10% or greater.



TestAmerica North Canton Certifications and Approvals:

California (#01144CA), Connecticut (#PH-0590), Florida (#E87225),

Illinois (#200004), Kansas (#E10336), Minnesota (#39-999-348), New Jersey (#OH001),

New York (#10975), Ohio VAP (#CL0024), West Virginia (#210), Wisconsin (#999518190), NAVY, ARMY, USDA Soil Permit

APPENDIX 2

EXAMPLE OF EXPANDED DELIVERABLE TRACKING SHEET

EXPANDED DELIVERABLE TRACKING SHEET

Lot Num.	PM	CLIENT	SDG	Analytical Due Date	Due Date	MS VOL		MSSEMI	
						IN	OUT	IN	OUT
A0F150169	ALM	57787	15169	29-Jun	5-Jul			6/23	7/6
A0L060216	DJP	371899	60216	27-Dec	3-Jan	12/18	12/20		
A0J060262	KJK	348218	348218	20-Oct	3-Nov	NO ED			
A0G100107	DJP	406511		2-Aug	11-Aug	7/21	8/2	7/31	8/3
A0K100231	JCS	405657		16-Nov	22-Nov			11/28	11/29

APPENDIX 3

EXAMPLE OF NARRATIVE CHECKLIST

NARRATIVE CHECKLIST

LOT NUMBER	DUE DATE	TO PA	TO PM	
A1C010105	3/21	3/5	Invoice only	
A1C010128	3/21	3/9	B 3/9	S 3/5
A1C010139	3/14	3/8	D 3/8	
A1C010145	3/23	3/6	D 3/6	
A1C010152	3/19	3/8	D 3/8	S 3/7
A1C010153	3/16	3/8	D 3/8	S 3/7
A1C010159	3/12	3/5	D3/5	

APPENDIX 4

WET CHEMISTRY PARAMETER RELATIONSHIP CHECKLIST

TestAmerica North Canton
General Chemistry Water Parameter Relationships

Lot #	Sample #	Client ID		
Alkalinity				
	Total mg/L	Bi- carbonate mg/L +	Carbonate mg/L +	Hydroxide mg/L
sample results	0	0	0	0
accuracy criteria	20%	20%	20%	20%
Alkalinity				Relationship Check OK --
Arsenic				
	Total As mg/L	As(III) mg/L +	As(V) mg/L	
sample results	0	0	0	
accuracy criteria	10%	30%	30%	
Arsenic				Relationship Check OK --
Chromium				
	Total Cr mg/L	Cr(VI) mg/L		
sample results	0	0		
accuracy criteria	10%	10%		
Chromium				Relationship Check OK --
Cyanide (CN)				
	Total ug/L	Free ug/L	Reactive ug/L	Amenable to chlorination ug/L
sample results	0	0	0	0
accuracy criteria	30%	30%	30%	30%
Cyanide				Relationship Check OK --
	Total ug/L	TCLP ug/L	SPLP ug/L	Deionized Water Leach ug/L
sample results	0	0	0	0
accuracy criteria	30%	30%	30%	30%
Cyanide				Relationship Check OK --
Demand				
	COD mg/L	BOD*1.5 mg/L		
sample results	0	0		
accuracy criteria	10%	30%		
Demand				Relationship Check OK --

TestAmerica North Canton
General Chemistry Water Parameter Relationships

<div> <div>COD mg/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>10%</div> </div>		≥	<div> <div>TOC mg/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>10%</div> </div>	Demand	OK --
Hexane Extractable Material (HEM) and Silica Gel Treated HEM (SGT-HEM)					
<div> <div>HEM mg/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>15%</div> </div>		≥	<div> <div>SGT-HEM mg/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>34%</div> </div>	HEM	OK --
Iron (Total iron and ferrous iron [Fe(II)])					
<div> <div>Total Fe mg/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>23%</div> </div>		≥	<div> <div>Fe(II) mg/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>10%</div> </div>	Fe	OK --
Nitrogen (N)					
<div> <div>Total Kjeldahl-N (TKN) ug/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>20%</div> <div>RLs 3</div> </div>		≥	<div> <div>Total Organic-N ug/L +</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>20%</div> <div>3</div> </div> <div> <div>Ammonia-N (NH3) ug/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>20%</div> <div>1 or 0.2</div> </div>	TKN	if NH3 < 3 relationship is OK OK --
<div> <div>Total NO₂ & NO₃ ug/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>15%</div> </div>		≥	<div> <div>NO₂ ug/L +</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>15%</div> </div> <div> <div>NO₃ ug/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>15%</div> </div>	NO ₂ &NO ₃	OK --
Phosphate (P)					
<div> <div>Total mg/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>25%</div> </div>		≥	<div> <div>Ortho P mg/L +</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>10%</div> </div> <div> <div>Organic mg/L +</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>25%</div> </div> <div> <div>Hydrolyzable mg/L</div> <div>sample results</div> <div>accuracy criteria</div> <div>0</div> <div>25%</div> </div>	Phosphate	OK --

**TestAmerica North Canton
 General Chemistry Water Parameter Relationships**

Solid content

sample results	Total Solids mg/L	0	≥	Total Dissolved Solids mg/L +	0	Total Suspended Solids mg/L	0	Total Solids	OK					
accuracy criteria	20%			20%		20%		--						
sample results	Total Dissolved Solids mg/L	0	≥	Total Alkalinity mg/L +	0	Total Hardness mg/L +	0	Chloride mg/L +	0	Sulfate mg/L	0	create observation NCM to indicate when matrix solids cause elevated RLs and likely cause of relationship failure	Total Dissolved Solids	OK
accuracy criteria	20%			20%		20%		10%		10%		--		
RL	10													
sample results	Total Dissolved Solids mg/L	0	≥	Conductivity uS	0			Total Dissolved Solids	OK					
accuracy criteria	20%			10%				--						
sample results	Total S ²⁻ mg/L	0	≥	Reactive S ²⁻ mg/L	0			Sulfide	OK					
accuracy criteria	20%			20%				--						

APPENDIX B

FIELD SOPs

FIELD S.O.P. NO. 1

SOIL SAMPLE COLLECTION PROCEDURE

Soil sampling will be conducted in accordance with EPA/540/P91/006 "Compendium of ERT Soil Sampling and Surface Geophysics Procedures" - Soil Sampling: SOP #2012 (USEPA 1991b), which is attached to this SOP.

1. Laboratory - supplied wide mouth jars will be used. Jars will be filled to the neck.
2. A unique numbering system will be used to identify each collected sample. This system will provide a tracking number to allow retrieval and cross-referencing of sample information. A listing of the sample identification numbers with written descriptions of sample location, type, time, and date will be maintained by the on-Site sampling personnel. The sample number system to be used is described as follows:

Example: S-YYMMDD-AA-XXX

Where:

S	designates sample type(S - soil)
YYMMDD	date of collection (year, month, day)
AA	sampler initials
XXX	sequential number starting with 001 for each event

QC samples will also be numbered with a unique location number using this numbering system.

The on-Site sampling personnel will be responsible for recording the sampling activities for each day and will record in the log book the following with respect to each sample:

- Unique sample identification number;
 - Sampling location identification;
 - Date/time of sample collection; and
 - Sampling data/remarks.
3. Samples will be placed on ice in laboratory-supplied coolers after collection. Sample labels may be covered with clear, waterproof tape. Cushioning material, such as

vermiculite, packing peanuts, or bubble wrap, will be placed around the sample containers in the cooler to protect them from breakage.

4. Chain-of-custody records will be used to track all samples from the time of sampling to the arrival of samples at the laboratory. Two original copies of the chain-of-custody record will accompany the sample shipment to the laboratory and will be signed and retained by the receiving laboratory's sample custodian. The chain-of-custody record will be retained by the shipper. One completed copy will be included in the final lab report by the laboratory. A typical chain-of-custody form, sample label and custody seal are attached to this SOP.
5. Samples will usually be shipped to the laboratory the same day the samples are collected in the field.

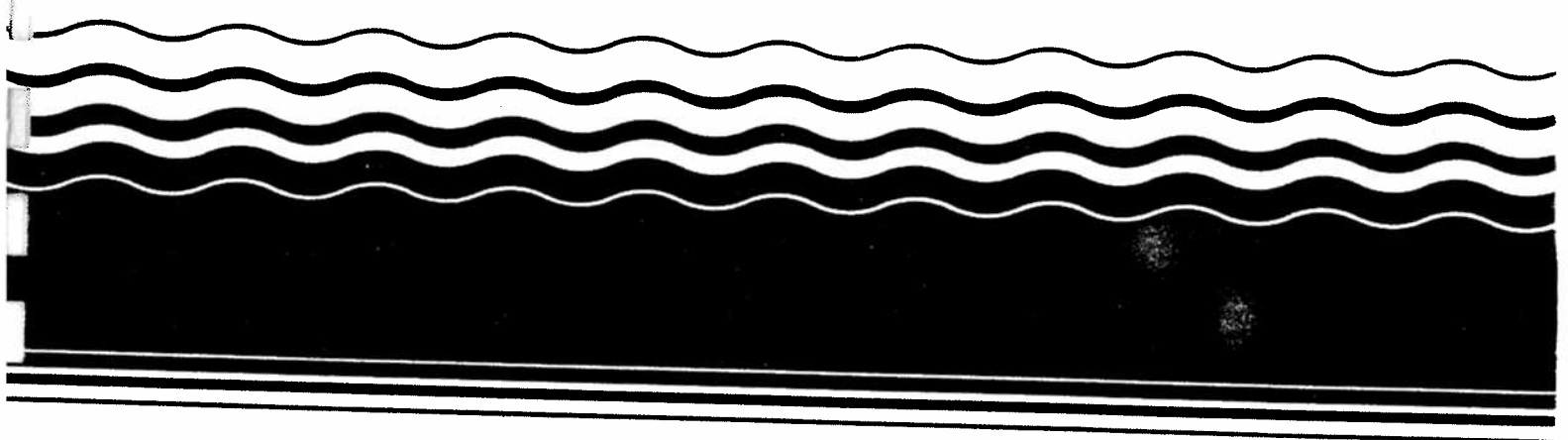
TestAmerica <small>THE LEADER IN ENVIRONMENTAL TESTING</small> 142954	Custody Seal <hr/> DATE <hr/> SIGNATURE	TestAmerica <small>THE LEADER IN ENVIRONMENTAL TESTING</small> 142954
--	--	--

CRA - Conestoga Rovers & Associates	
Job #:	Date:
Job Name:	Sampler:
Sample I.D.:	
Analysis:	
Notes:	

Sample label



Compendium of ERT Soil Sampling and Surface Geophysics Procedures



COMPENDIUM OF ERT SOIL SAMPLING AND SURFACE GEOPHYSICS PROCEDURES

Sampling Equipment Decontamination

Soil Sampling

Soil Gas Sampling

General Surface Geophysics

Interim Final

Environmental Response Team
Emergency Response Division

Office of Emergency and Remedial Response
U.S. Environmental Protection Agency
Washington, DC 20460



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The policies and procedures established in this document are intended solely for the guidance of government personnel for use in the Superfund Removal Program. They are not intended, and cannot be relied upon, to create any rights, substantive or procedural, enforceable by any party in litigation with the United States. The Agency reserves the right to act at variance with these policies and procedures and to change them at any time without public notice.

Depending on circumstances and needs, it may not be possible or appropriate to follow these procedures exactly in all situations due to site conditions, equipment limitations, and limitations of the standard procedures. Whenever these procedures cannot be followed as written, they may be used as general guidance with any and all modifications fully documented in either QA Plans, Sampling Plans, or final reports of results.

Each Standard Operating Procedure in this compendium contains a discussion on quality assurance/quality control (QA/QC). For more information on QA/QC objectives and requirements, refer to the *Quality Assurance/Quality Control Guidance for Removal Activities*, OSWER directive 9360.4-01, EPA/540/G-90/004.

Questions, comments, and recommendations are welcomed regarding the Compendium of ERT Soil Sampling and Surface Geophysics Procedures. Send remarks to:

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National Technical Information Service (NTIS)
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1.0 SAMPLING EQUIPMENT DECONTAMINATION: SOP #2006

1.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes methods used for preventing or reducing cross-contamination, and provides general guidelines for sampling equipment decontamination procedures at a hazardous waste site. Preventing or minimizing cross-contamination in sampled media and in samples is important for preventing the introduction of error into sampling results and for protecting the health and safety of site personnel.

Removing or neutralizing contaminants that have accumulated on sampling equipment ensures protection of personnel from permeating substances, reduces or eliminates transfer of contaminants to clean areas, prevents the mixing of incompatible substances, and minimizes the likelihood of sample cross-contamination.

1.2 METHOD SUMMARY

Contaminants can be physically removed from equipment, or deactivated by sterilization or disinfection. Gross contamination of equipment requires physical decontamination, including abrasive and non-abrasive methods. These include the use of brushes, air and wet blasting, and high-pressure water cleaning, followed by a wash/rinse process using appropriate cleaning solutions. Use of a solvent rinse is required when organic contamination is present.

1.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

This section is not applicable to this SOP.

1.4 INTERFERENCES AND POTENTIAL PROBLEMS

- The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment

provided that it has been verified by laboratory analysis to be analyte free.

- An untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal water treatment system for mixing of decontamination solutions.
- Acids and solvents utilized in the decontamination sequence pose the health and safety risks of inhalation or skin contact, and raise shipping concerns of permeation or degradation.
- The site work plan must address disposal of the spent decontamination solutions.
- Several procedures can be established to minimize contact with waste and the potential for contamination. For example:
 - Stress work practices that minimize contact with hazardous substances.
 - Use remote sampling, handling, and container-opening techniques when appropriate.
 - Cover monitoring and sampling equipment with protective material to minimize contamination.
 - Use disposable outer garments and disposable sampling equipment when appropriate.

1.5 EQUIPMENT/APPARATUS

- appropriate personal protective clothing
- non-phosphate detergent
- selected solvents
- long-handled brushes
- drop cloths/plastic sheeting
- trash container
- paper towels
- galvanized tubs or buckets
- tap water

- distilled/deionized water
- metal/plastic containers for storage and disposal of contaminated wash solutions
- pressurized sprayers for tap and deionized/distilled water
- sprayers for solvents
- trash bags
- aluminum foil
- safety glasses or splash shield
- emergency eyewash bottle

1.6 REAGENTS

There are no reagents used in this procedure aside from the actual decontamination solutions and solvents. In general, the following solvents are utilized for decontamination purposes:

- 10% nitric acid⁽¹⁾
- acetone (pesticide grade)⁽²⁾
- hexane (pesticide grade)⁽²⁾
- methanol

⁽¹⁾ Only if sample is to be analyzed for trace metals.

⁽²⁾ Only if sample is to be analyzed for organics.

1.7 PROCEDURES

As part of the health and safety plan, develop and set up a decontamination plan before any personnel or equipment enter the areas of potential exposure. The equipment decontamination plan should include:

- the number, location, and layout of decontamination stations
- which decontamination apparatus is needed
- the appropriate decontamination methods
- methods for disposal of contaminated clothing, apparatus, and solutions

1.7.1 Decontamination Methods

All personnel, samples, and equipment leaving the contaminated area of a site must be decontaminated. Various decontamination methods will either physically remove contaminants, inactivate contaminants by disinfection or sterilization, or do both.

In many cases, gross contamination can be removed by physical means. The physical decontamination techniques appropriate for equipment decontamination can be grouped into two categories: abrasive methods and non-abrasive methods.

Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing and wearing away the top layer of the surface containing the contaminant. The following abrasive methods are available:

- **Mechanical:** Mechanical cleaning methods use brushes of metal or nylon. The amount and type of contaminants removed will vary with the hardness of bristles, length of brushing time, and degree of brush contact.
- **Air Blasting:** Air blasting is used for cleaning large equipment, such as bulldozers, drilling rigs or auger bits. The equipment used in air blast cleaning employs compressed air to force abrasive material through a nozzle at high velocities. The distance between the nozzle and the surface cleaned, as well as the pressure of air, the time of application, and the angle at which the abrasive strikes the surface, determines cleaning efficiency. Air blasting has several disadvantages: it is unable to control the amount of material removed, it can aerate contaminants, and it generates large amounts of waste.
- **Wet Blasting:** Wet blast cleaning, also used to clean large equipment, involves use of a suspended fine abrasive delivered by compressed air to the contaminated area. The amount of materials removed can be carefully controlled by using very fine abrasives. This method generates a large amount of waste.

Non-Abrasive Cleaning Methods

Non-abrasive cleaning methods work by forcing the contaminant off of a surface with pressure. In general, less of the equipment surface is removed using non-abrasive methods. The following non-abrasive methods are available:

- **High-Pressure Water:** This method consists of a high-pressure pump, an operator-controlled directional nozzle, and a high pressure hose. Operating pressure usually ranges from 340 to 680 atmospheres (atm) which relates to flow rates of 20 to 140 liters per minute.
- **Ultra-High-Pressure Water:** This system produces a pressurized water jet (from 1,000 to 4,000 atm). The ultra-high-pressure spray removes tightly-adhered surface film. The water velocity ranges from 500 m/sec (1,000 atm) to 900 m/sec (4,000 atm). Additives can enhance the method. This method is not applicable for hand-held sampling equipment.

Disinfection/Rinse Methods

- **Disinfection:** Disinfectants are a practical means of inactivating infectious agents.
- **Sterilization:** Standard sterilization methods involve heating the equipment. Sterilization is impractical for large equipment.
- **Rinsing:** Rinsing removes contaminants through dilution, physical attraction, and solubilization.

1.7.2 Field Sampling Equipment Cleaning Procedures

Solvent rinses are not necessarily required when organics are not a contaminant of concern and may be eliminated from the sequence specified below. Similarly, an acid rinse is not required if analysis does not include inorganics.

1. Where applicable, follow physical removal procedures specified in section 1.7.1.
2. Wash equipment with a non-phosphate detergent solution.
3. Rinse with tap water.
4. Rinse with distilled/deionized water.
5. Rinse with 10% nitric acid if the sample will be analyzed for trace organics.

6. Rinse with distilled/deionized water.
7. Use a solvent rinse (pesticide grade) if the sample will be analyzed for organics.
8. Air dry the equipment completely.
9. Rinse again with distilled/deionized water.

Selection of the solvent for use in the decontamination process is based on the contaminants present at the site. Use of a solvent is required when organic contamination is present on-site. Typical solvents used for removal of organic contaminants include acetone, hexane, or water. An acid rinse step is required if metals are present on-site. If a particular contaminant fraction is not present at the site, the nine-step decontamination procedure listed above may be modified for site specificity. The decontamination solvent used should not be among the contaminants of concern at the site.

Table 1 lists solvent rinses which may be required for elimination of particular chemicals. After each solvent rinse, the equipment should be air dried and rinsed with distilled/deionized water.

Sampling equipment that requires the use of plastic tubing should be disassembled and the tubing replaced with clean tubing, before commencement of sampling and between sampling locations.

1.8 CALCULATIONS

This section is not applicable to this SOP.

1.9 QUALITY ASSURANCE/ QUALITY CONTROL

One type of quality control sample specific to the field decontamination process is the rinsate blank. The rinsate blank provides information on the effectiveness of the decontamination process employed in the field. When used in conjunction with field blanks and trip blanks, a rinsate blank can detect contamination during sample handling, storage and sample transportation to the laboratory.

Table 1: Recommended Solvent Rinse for Soluble Contaminants

SOLVENT	SOLUBLE CONTAMINANTS
Water	<ul style="list-style-type: none"> • Low-chain hydrocarbons • Inorganic compounds • Salts • Some organic acids and other polar compounds
Dilute Acids	<ul style="list-style-type: none"> • Basic (caustic) compounds • Amines • Hydrazines
Dilute Bases -- for example, detergent and soap	<ul style="list-style-type: none"> • Metals • Acidic compounds • Phenol • Thiols • Some nitro and sulfonic compounds
Organic Solvents ⁽¹⁾ - for example, alcohols, ethers, ketones, aromatics, straight-chain alkanes (e.g., hexane), and common petroleum products (e.g., fuel, oil, kerosene)	<ul style="list-style-type: none"> • Nonpolar compounds (e.g., some organic compounds)

⁽¹⁾ - WARNING: Some organic solvents can permeate and/or degrade protective clothing.

A rinsate blank consists of a sample of analyte-free (i.e, deionized) water which is passed over and through a field decontaminated sampling device and placed in a clean sample container.

Rinsate blanks should be run for all parameters of interest at a rate of 1 per 20 for each parameter, even if samples are not shipped that day. Rinsate blanks are not required if dedicated sampling equipment is used.

1.10 DATA VALIDATION

This section is not applicable to this SOP.

1.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA and specific health and safety procedures.

Decontamination can pose hazards under certain circumstances even though performed to protect

health and safety. Hazardous substances may be incompatible with decontamination methods. For example, the decontamination solution or solvent may react with contaminants to produce heat, explosion, or toxic products. Decontamination methods may be incompatible with clothing or equipment; some solvents can permeate or degrade protective clothing. Also, decontamination solutions and solvents may pose a direct health hazard to workers through inhalation or skin contact, or if they combust.

The decontamination solutions and solvents must be determined to be compatible before use. Any method that permeates, degrades, or damages personal protective equipment should not be used. If decontamination methods pose a direct health hazard, measures should be taken to protect personnel or the methods should be modified to eliminate the hazard.

2.0 SOIL SAMPLING: SOP #2012

2.1 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures for collecting representative soil samples. Analysis of soil samples may determine whether concentrations of specific soil pollutants exceed established action levels, or if the concentrations of soil pollutants present a risk to public health, welfare, or the environment.

2.2 METHOD SUMMARY

Soil samples may be collected using a variety of methods and equipment. The methods and equipment used are dependent on the depth of the desired sample, the type of sample required (disturbed versus undisturbed), and the type of soil. Near-surface soils may be easily sampled using a spade, trowel, and scoop. Sampling at greater depths may be performed using a hand auger, a trier, a split-spoon, or, if required, a backhoe.

2.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Chemical preservation of solids is not generally recommended. Refrigeration to 4°C, supplemented by a minimal holding time, is usually the best approach.

2.4 INTERFERENCES AND POTENTIAL PROBLEMS

There are two primary interferences or potential problems associated with soil sampling. These include cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, disturbance of the matrix resulting in compaction of the sample, or inadequate homogenization of the samples where required,

resulting in variable, non-representative results.

2.5 EQUIPMENT/APPARATUS

- sampling plan
- maps/plot plan
- safety equipment, as specified in the health and safety plan
- compass
- tape measure
- survey stakes or flags
- camera and film
- stainless steel, plastic, or other appropriate homogenization bucket or bowl
- 1-quart mason jars w/Teflon liners
- Ziploc plastic bags
- logbook
- labels
- chain of custody forms and seals
- field data sheets
- cooler(s)
- ice
- decontamination supplies/equipment
- canvas or plastic sheet
- spade or shovel
- spatula
- scoop
- plastic or stainless steel spoons
- trowel
- continuous flight (screw) auger
- bucket auger
- post hole auger
- extension rods
- T-handle
- sampling trier
- thin-wall tube sampler
- Vehimeyer soil sampler outfit
 - tubes
 - points
 - drive head
 - drop hammer
 - puller jack and grip
- backhoe

2.6 REAGENTS

Reagents are not used for the preservation of soil samples. Decontamination solutions are specified in

2.7 PROCEDURES

2.7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and which equipment and supplies are required.
2. Obtain necessary sampling and monitoring equipment.
3. Decontaminate or pre-clean equipment, and ensure that it is in working order.
4. Prepare schedules, and coordinate with staff, client, and regulatory agencies, if appropriate.
5. Perform a general site survey prior to site entry in accordance with the site-specific health and safety plan.
6. Use stakes, buoys, or flagging to identify and mark all sampling locations. Consider specific site factors, including extent and nature of contaminant, when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations will be utility-cleared by the property owner prior to soil sampling.

2.7.2 Sample Collection

Surface Soil Samples

Collect samples from near-surface soil with tools such as spades, shovels, and scoops. Surface material can be removed to the required depth with this equipment, then a stainless steel or plastic scoop can be used to collect the sample.

This method can be used in most soil types but is limited to sampling near surface areas. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sampling team member. The use of a flat, pointed mason trowel to cut a block of the desired soil can be helpful when undisturbed profiles are required. A stainless steel scoop, lab spoon, or plastic spoon will suffice in most other

applications. Avoid the use of devices plated with chrome or other materials. Plating is particularly common with garden implements such as potting trowels.

Follow these procedures to collect surface soil samples.

1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.
3. If volatile organic analysis is to be performed, transfer a portion of the sample directly into an appropriate, labeled sample container(s) with a stainless steel lab spoon, plastic lab spoon, or equivalent and secure the cap(s) tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into an appropriate, labeled container(s) and secure the cap(s) tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled container(s) and secure the cap(s) tightly.

Sampling at Depth with Augers and Thin-Wall Tube Samplers

This system consists of an auger, a series of extensions, a "T" handle, and a thin-wall tube sampler (Appendix A, Figure 1). The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin-wall tube sampler. The system is then lowered down the borehole, and driven into the soil at the completion depth. The system is withdrawn and the core collected from the thin-wall tube sampler.

Several types of augers are available. These include: bucket, continuous flight (screw), and posthole augers. Bucket augers are better for direct

sample recovery since they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights, which are usually at 5-foot intervals. The continuous flight augers are satisfactory for use when a composite of the complete soil column is desired. Posthole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil.

Follow these procedures for collecting soil samples with the auger and a thin-wall tube sampler.

1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod.
2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first 3 to 6 inches of surface soil for an area approximately 6 inches in radius around the drilling location.
3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.
4. After reaching the desired depth, slowly and carefully remove the auger from boring. When sampling directly from the auger, collect sample after the auger is removed from boring and proceed to Step 10.
5. Remove auger tip from drill rods and replace with a pre-cleaned thin-wall tube sampler. Install proper cutting tip.
6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring as the vibrations may cause the boring walls to collapse.
7. Remove the tube sampler, and unscrew the drill rods.
8. Remove the cutting tip and the core from the device.

9. Discard the top of the core (approximately 1 inch), as this represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container(s). Sample homogenization is not required.
10. If volatile organic analysis is to be performed, transfer a portion of the sample directly into an appropriate, labeled sample container(s) with a stainless steel lab spoon, plastic lab spoon, or equivalent and secure the cap(s) tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into an appropriate, labeled container(s) and secure the cap(s) tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into the appropriate, labeled container(s) and secure the cap(s) tightly.
11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.
12. Abandon the hole according to applicable state regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

Sampling at Depth with a Trier

The system consists of a trier, and a "T" handle. The auger is driven into the soil to be sampled and used to extract a core sample from the appropriate depth.

Follow these procedures to collect soil samples with a sampling trier.

1. Insert the trier (Appendix A, Figure 2) into the material to be sampled at a 0° to 45° angle from horizontal. This orientation minimizes the spillage of sample.
2. Rotate the trier once or twice to cut a core of material.

3. Slowly withdraw the trier, making sure that the slot is facing upward.
4. If volatile organic analysis is to be performed, transfer a portion of the sample directly into an appropriate, labeled sample container(s) with a stainless steel lab spoon, plastic lab spoon, or equivalent and secure the cap(s) tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into an appropriate, labeled container(s) and secure the cap(s) tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into an appropriate, labeled container(s) and secure the cap(s) tightly.

Sampling at Depth with a Split Spoon (Barrel) Sampler

The procedure for split spoon sampling describes the collection and extraction of undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

When split tube sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D 1586-67 (reapproved 1974).

Follow these procedures for collecting soil samples with a split spoon.

1. Assemble the sampler by aligning both sides of the barrel and then screwing the bit onto the bottom and the heavier head piece onto the top.
2. Place the sampler in a perpendicular position on the sample material.
3. Using a sledge hammer or well ring, if available, drive the tube. Do not drive past the bottom of the head piece or compression of the

sample will result.

4. Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.
5. Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in diameters of 2 and 3 1/2 inches. However, in order to obtain the required sample volume, use of a larger barrel may be required.
6. Without disturbing the core, transfer it to an appropriate labeled sample container(s) and seal tightly.

Test Pit/Trench Excavation

These relatively large excavations are used to remove sections of soil, when detailed examination of soil characteristics (horizontal structure, color, etc.) are required. It is the least cost effective sampling method due to the relatively high cost of backhoe operation.

Follow these procedures for collecting soil samples from test pit/trench excavations.

1. Prior to any excavation with a backhoe, it is important to ensure that all sampling locations are clear of utility lines and poles (subsurface as well as above surface).
2. Using the backhoe, dig a trench to approximately 3 feet in width and approximately 1 foot below the cleared sampling location. Place removed or excavated soils on plastic sheets. Trenches greater than 5 feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.
3. Use a shovel to remove a 1- to 2-inch layer of soil from the vertical face of the pit where sampling is to be done.
4. Take samples using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling

to remove any soil that may have fallen from above, and to expose fresh soil for sampling. In many instances, samples can be collected directly from the backhoe bucket.

5. If volatile organic analysis is to be performed, transfer a portion of the sample directly into an appropriate, labeled sample container(s) with a stainless steel lab spoon, plastic lab spoon, or equivalent and secure the cap(s) tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into an appropriate, labeled container(s) and secure the cap(s) tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled container(s) and secure the cap(s) tightly.
6. Abandon the pit or excavation according to applicable state regulations. Generally, shallow excavations can simply be backfilled with the removed soil material.

2.8 CALCULATIONS

This section is not applicable to this SOP.

2.9 QUALITY ASSURANCE/ QUALITY CONTROL

There are no specific quality assurance activities which apply to the implementation of these procedures. However, the following QA procedures apply:

- All data must be documented on field data sheets or within site logbooks.
- All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

2.10 DATA VALIDATION

This section is not applicable to this SOP.

2.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and specific health and safety procedures.

3.0 SOIL GAS SAMPLING: SOP #2149

3.1 SCOPE AND APPLICATION

Soil gas monitoring provides a quick means of waste site evaluation. Using this method, underground contamination can be identified, and the source, extent, and movement of the pollutants can be traced.

This Standard Operating Procedure (SOP) outlines the methods used by EPA/ERT in installing soil gas wells; measuring organic levels in the soil gas using an HNU PI 101 Portable Photoionization Analyzer and/or other air monitoring devices; and sampling the soil gas using Tedlar bags, Tenax sorbent tubes, and SUMMA canisters.

3.2 METHOD SUMMARY

A 3/8-inch diameter hole is driven into the ground to a depth of 4 to 5 feet using a commercially available "slam bar". (Soil gas can also be sampled at other depths by the use of a longer bar or bar attachments.) A 1/4-inch O.D. stainless steel probe is inserted into the hole. The hole is then sealed at the top around the probe using modeling clay. The gas contained in the interstitial spaces of the soil is sampled by pulling the sample through the probe using an air sampling pump. The sample may be stored in Tedlar bags, drawn through sorbent cartridges, or analyzed directly using a direct reading instrument.

The air sampling pump is not used for SUMMA canister sampling of soil gas. Sampling is achieved by soil gas equilibration with the evacuated SUMMA canister. Other field air monitoring devices, such as the combustible gas indicator (MSA CGI/02 Meter, Model 260) and the organic vapor analyzer (Foxboro OVA, Model 128), can also be used depending on specific site conditions. Measurement of soil temperature using a temperature probe may also be desirable. Bagged samples are usually analyzed in a field laboratory using a portable Photovac GC.

Power driven sampling probes may be utilized when soil conditions make sampling by hand unfeasible (i.e., frozen ground, very dense clays, pavement,

etc.). Commercially available soil gas sampling probes (hollow, 1/2-inch O.D. steel probes) can be driven to the desired depth using a power hammer (e.g., Bosch Demolition Hammer). Samples can be drawn through the probe itself, or through Teflon tubing inserted through the probe and attached to the probe point. Samples are collected and analyzed as described above.

3.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.3.1 Tedlar Bag

Soil gas samples are generally contained in 1-L Tedlar bags. Bagged samples are best stored in coolers to protect the bags from any damage that may occur in the field or in transit. In addition, coolers ensure the integrity of the samples by keeping them at a cool temperature and out of direct sunlight. Samples should be analyzed as soon as possible, preferably within 24 to 48 hours.

3.3.2 Tenax Tube

Bagged samples can also be drawn into Tenax or other sorbent tubes to undergo lab GC/MS analysis. If Tenax tubes are to be utilized, special care must be taken to avoid contamination. Handling of the tubes should be kept to a minimum, and samplers must wear nylon or other lint-free gloves. After sampling, each tube should be stored in a clean, sealed culture tube; the ends packed with clean glass wool to protect the sorbent tube from breakage. The culture tubes should be kept cool and wrapped in aluminum foil to prevent any photodegradation of samples (see Section 3.7.4.).

3.3.3 SUMMA Canister

The SUMMA canisters used for soil gas sampling have a 6-L sample capacity and are certified clean by GC/MS analysis before being utilized in the field. After sampling is completed, they are stored and shipped in travel cases.

3.4 INTERFERENCES AND POTENTIAL PROBLEMS

3.4.1 HNU Measurements

A number of factors can affect the response of the HNU PI 101. High humidity can cause lamp fogging and decreased sensitivity. This can be significant when soil moisture levels are high, or when a soil gas well is actually in groundwater. High concentrations of methane can cause a downscale deflection of the meter. High and low temperature, electrical fields, FM radio transmission, and naturally occurring compounds, such as terpenes in wooded areas, will also affect instrument response.

Other field screening instruments can be affected by interferences. Consult the manufacturers' manuals.

3.4.2 Factors Affecting Organic Concentrations in Soil Gas

Concentrations in soil gas are affected by dissolution, adsorption, and partitioning. Partitioning refers to the ratio of component found in a saturated vapor above an aqueous solution to the amount in the solution; this can, in theory, be calculated using the Henry's Law constants. Contaminants can also be adsorbed onto inorganic soil components or "dissolved" in organic components. These factors can result in a lowering of the partitioning coefficient.

Soil "tightness" or amount of void space in the soil matrix, will affect the rate of recharging of gas into the soil gas well.

Existence of a high, or perched, water table, or of an impermeable underlying layer (such as a clay lens or layer of buried slag) may interfere with sampling of the soil gas. Knowledge of site geology is useful in such situations, and can prevent inaccurate sampling.

3.4.3 Soil Probe Clogging

A common problem with this sampling method is soil probe clogging. A clogged probe can be identified by using an in-line vacuum gauge or by listening for the sound of the pump laboring. This problem can usually be eliminated by using a wire cable to clear the probe (see procedure #3 in Section 3.7.1).

3.4.4 Underground Utilities

Prior to selecting sample locations, an underground utility search is recommended. The local utility companies can be contacted and requested to mark the locations of their underground lines. Sampling plans can then be drawn up accordingly. Each sample location should also be screened with a metal detector or magnetometer to verify that no underground pipes or drums exist.

3.5 EQUIPMENT/APPARATUS

3.5.1 Slam Bar Method

- slam bar (one per sampling team)
- soil gas probes, stainless steel tubing, 1/4-inch O.D., 5 foot length
- flexible wire or cable used for clearing the tubing during insertion into the well
- "quick connect" fittings to connect sampling probe tubing, monitoring instruments, and Gilian pumps to appropriate fittings on vacuum box
- modeling clay
- vacuum box for drawing a vacuum around Tedlar bag for sample collection (one per sampling team)
- Gilian pump Model HFS113A adjusted to approximately 3.0 L/min (one to two per sampling team)
- 1/4-inch Teflon tubing, 2 to 3 foot lengths, for replacement of contaminated sample line
- Tedlar bags, 1 liter, at least one bag per sample point
- soil gas sampling labels, field data sheets, logbook, etc.
- HNU Model PI 101, or other field air monitoring devices, (one per sampling team)
- ice chest, for carrying equipment and for protection of samples (two per sampling team)
- metal detector or magnetometer, for detecting underground utilities/pipes/drums (one per sampling team)
- Photovac GC, for field-lab analysis of bagged samples
- SUMMA canisters (plus their shipping cases) for sample, storage and transportation

3.5.2 Power Hammer Method

- Bosch demolition hammer
- 1/2-inch O.D. steel probes, extensions, and points
- dedicated aluminum sampling points
- Teflon tubing, 1/4-inch O.D.
- "quick connect" fittings to connect sampling probe tubing, monitoring instruments, and Gilian pumps to appropriate fittings on vacuum box
- modeling clay
- vacuum box for drawing a vacuum around Tedlar bag for sample collection (one per sampling team)
- Gilian pump Model HFS113A adjusted to approximately 3.0 L/min (one to two per sampling team)
- 1/4-inch Teflon tubing, 2 to 3 foot lengths, for replacement of contaminated sample line
- Tedlar bags, 1 liter, at least one bag per sample point
- soil gas sampling labels, field data sheets, logbook, etc.
- HNU Model PI 101, or other field air monitoring devices, (one per sampling team)
- ice chest, for carrying equipment and for protection of samples (two per sampling team)
- metal detector or magnetometer, for detecting underground utilities/pipes/drums (one per sampling team)
- Photovac GC, for field-lab analysis of bagged samples
- SUMMA canisters (plus their shipping cases) for sample, storage and transportation
- generator with extension cords
- high lift jack assembly for removing probes

3.6 REAGENTS

- HNU Systems Inc. Calibration Gas for HNU Model PI 101, and/or calibration gas for other field air monitoring devices
- deionized organic-free water, for decontamination
- methanol, HPLC grade, for decontamination
- ultra-zero grade compressed air, for field blanks

- standard gas preparations for Photovac GC calibration and Tedlar bag spikes

3.7 PROCEDURES

3.7.1 Soil Gas Well Installation

1. Initially, make a hole slightly deeper than the desired depth. For sampling up to 5 feet, use a 5-foot single piston slam bar. For deeper depths, use a piston slam bar with threaded 4-foot-long extensions. Other techniques can be used, so long as holes are of narrow diameter and no contamination is introduced.
2. After the hole is made, carefully withdraw the slam bar to prevent collapse of the walls of the hole. Then insert the soil gas probe.
3. It is necessary to prevent plugging of the probe, especially for deeper holes. Place a metal wire or cable, slightly longer than the probe, into the probe prior to inserting into the hole. Insert the probe to full depth, then pull it up 3 to 6 inches, then clear it by moving the cable up and down. The cable is removed before sampling.
4. Seal the top of the sample hole at the surface against ambient air infiltration by using modeling clay molded around the probe at the surface of the hole.
5. If conditions preclude hand installation of the soil gas wells, the power driven system may be employed. Use the generator-powered demolition hammer to drive the probe to the desired depth (up to 12 feet may be attained with extensions). Pull the probe up 1 to 3 inches if the retractable point is used. No clay is needed to seal the hole. After sampling, retrieve the probe using the high lift jack assembly.
6. If semi-permanent soil gas wells are required, use the dedicated aluminum probe points. Insert these points into the bottom of the power-driven probe and attach it to the Teflon tubing. Insert the probe as in step 5. When the probe is removed, the point and Teflon tube remain in the hole, which may be sealed by backfilling with sand, bentonite, or soil.

3.7.2 Screening with Field Instruments

1. The well volume **must** be evacuated prior to sampling. Connect the Gilian pump, adjusted to 3.0 L/min, to the sample probe using a section of Teflon tubing as a connector. Turn the pump on, and a vacuum is pulled through the probe for approximately 15 seconds. A longer time is required for sample wells of greater depths.
2. After evacuation, connect the monitoring instrument(s) to the probe using a Teflon connector. When the reading is stable, or peaks, record the reading. For detailed procedures on HNU field protocol, see appendix B, and refer to the manufacturer's instructions.
3. Some readings may be above or below the range set on the field instruments. The range may be reset, or the response recorded as a figure greater than or less than the range. Consider the recharge rate of the well with soil gas when sampling at a different range setting.

3.7.3 Tedlar Bag Sampling

1. Follow step 1 in section 3.7.2 to evacuate well volume. If air monitoring instrument screening was performed prior to sampling, evacuation is not necessary.
2. Use the vacuum box and sampling train (Figure 3 in Appendix A) to take the sample. The sampling train is designed to minimize the introduction of contaminants and losses due to adsorption. All wetted parts are either Teflon or stainless steel. The vacuum is drawn indirectly to avoid contamination from sample pumps.
3. Place the Tedlar bag inside the vacuum box, and attach it to the sampling port. Attach the sample probe to the sampling port via Teflon tubing and a "quick connect" fitting.
4. Draw a vacuum around the outside of the bag, using a Gilian pump connected to the vacuum box evacuation port, via Tygon tubing and a "quick connect" fitting. The vacuum causes the bag to inflate, drawing the sample.

5. Break the vacuum by removing the Tygon line from the pump. Remove the bagged sample from the box and close valve. Label bag, record data on data sheets or in logbooks. Record the date, time, sample location ID, and the HNU, or other instrument reading(s) on sample bag label.

CAUTION: Labels should not be pasted directly onto the bags, nor should bags be labeled directly using a marker or pen. Inks and adhesive may diffuse through the bag material, contaminating the sample. Place labels on the edge of the bags, or tie the labels to the metal eyelets provided on the bags. Markers with inks containing volatile organics (i.e., permanent ink markers) should not be used.

3.7.4 Tenax Tube Sampling

Samples collected in Tedlar bags may be sorbed onto Tenax tubes for further analysis by GC/MS.

Additional Apparatus

- Syringe with a luer-lock tip capable of drawing a soil gas or air sample from a Tedlar bag onto a Tenax/CMS sorbent tube. The syringe capacity is dependent upon the volume of sample being drawn onto the sorbent tube.
- Adapters for fitting the sorbent tube between the Tedlar bag and the sampling syringe. The adapter attaching the Tedlar bag to the sorbent tube consists of a reducing union (1/4-inch to 1/16-inch O.D. -- Swagelok cat. # SS-400-6-ILV or equivalent) with a length of 1/4-inch O.D. Teflon tubing replacing the nut on the 1/6-inch (Tedlar bag) side. A 1/4-inch I.D. silicone O-ring replaces the ferrules in the nut on the 1/4-inch (sorbent tube) side of the union.

The adapter attaching the sampling syringe to the sorbent tube consists of a reducing union (1/4-inch to 1/16-inch O.D. -- Swagelok Cat. # SS-400-6-ILV or equivalent) with a 1/4-inch I.D. silicone O-ring replacing the ferrules in the nut on the 1/4-inch (sorbent tube) side and the needle of a luer-lock syringe needle inserted into the 1/16-inch side (held in place with a 1/16-inch ferrule). The

luer-lock end of the needle can be attached to the sampling syringe. It is useful to have a luer-lock on/off valve situated between the syringe and the needle.

- Two-stage glass sampling cartridge (1/4-inch O.D. x 1/8-inch I.D. x 5 1/8 inch) contained in a flame-sealed tube (manufactured by Supelco Custom Tenax/Spherocarb Tubes or equivalent) containing two sorbent sections retained by glass wool:

Front section: 150 mg of Tenax-GC

Back section: 150 mg of CMS
(Carbonized Molecular Sieve)

Sorbent tubes may also be prepared in the lab and stored in either Teflon-capped culture tubes or stainless steel tube containers. Sorbent tubes stored in this manner should not be kept more than 2 weeks without reconditioning. (See SOP #2052 for Tenax/CMS sorbent tube preparation).

- Teflon-capped culture tubes or stainless steel tube containers for sorbent tube storage. These containers should be conditioned by baking at 120°C for at least 2 hours. The culture tubes should contain a glass wool plug to prevent sorbent tube breakage during transport. Reconditioning of the containers should occur between usage or after extended periods of disuse (i.e., 2 weeks or more).
- Nylon gloves or lint-free cloth. (Hewlett Packard Part # 8650-0030 or equivalent.)

Sample Collection

1. Handle sorbent tubes with care, using nylon gloves (or other lint-free material) to avoid contamination.
2. Immediately before sampling, break one end of the sealed tube and remove the Tenax cartridge. For in-house prepared tubes, remove cartridge from its container.
3. Connect the valve on the Tedlar bag to the sorbent tube adapter. Connect the sorbent tube to the sorbent tube adapter with the Tenax (white granular) side of the tube facing the Tedlar bag.
4. Connect the sampling syringe assembly to the CMS (black) side of the sorbent tube. Fittings on the adapters should be very tight.
5. Open the valve on the Tedlar bag.
6. Open the on/off valve of the sampling syringe.
7. Draw a predetermined volume of sample onto the sorbent tube. (This may require closing the syringe valve, emptying the syringe and then repeating the procedure, depending upon the syringe capacity and volume of sample required.)
8. After sampling, remove the tube from the sampling train with gloves or a clean cloth. Do not label or write on the Tenax/CMS tube.
9. Place the sorbent tube in a conditioned stainless steel tube holder or culture tube. Culture tube caps should be sealed with Teflon tape.

Sample Labeling

Each sample tube container (not tube) must be labeled with the site name, sample station number, sample date, and sample volume.

Chain of custody forms must accompany all samples to the laboratory.

Quality Assurance

Before field use, a QA check should be performed on each batch of sorbent tubes by analyzing a tube with thermal desorption/cryogenic trapping GC/MS.

At least one blank sample must be submitted with each set of samples collected at a site. This trip blank must be treated the same as the sample tubes except no sample will be drawn through the tube.

Sample tubes should be stored out of UV light (i.e., sunlight) and kept on ice until analysis.

Samples should be taken in duplicate, when possible.

3.7.5 SUMMA Canister Sampling

1. Follow item 1 in step 3.7.2 to evacuate well volume. If HNU analysis was performed prior to taking a sample, evacuation is not necessary.
2. Attach a certified clean, evacuated 6-L SUMMA canister via the 1/4-inch Teflon tubing.
3. Open the valve on SUMMA canister. The soil gas sample is drawn into the canister by pressure equilibration. The approximate sampling time for a 6-L canister is 20 minutes.
4. Site name, sample location, number, and date must be recorded on a chain of custody form and on a blank tag attached to the canister.

3.8 CALCULATIONS

3.8.1 Field Screening Instruments

Instrument readings are usually read directly from the meter. In some cases, the background level at the soil gas station may be subtracted:

$$\text{Final Reading} = \text{Sample Reading} - \text{Background}$$

3.8.2 Photovac GC Analysis

Calculations used to determine concentrations of individual components by Photovac GC analysis are beyond the scope of this SOP and are covered in ERT SOP #2109, Photovac GC Analysis for Soil, Water and Air/Soil Gas.

3.9 QUALITY ASSURANCE/ QUALITY CONTROL

3.9.1 Field Instrument Calibration

Consult the manufacturers' manuals for correct use and calibration of all instrumentation. The HNU should be calibrated at least once a day.

3.9.2 Gillan Model HFS113A Air Sampling Pump Calibration

Flow should be set at approximately 3.0 L/min;

accurate flow adjustment is not necessary. Pumps should be calibrated prior to bringing into the field.

3.9.3 Sample Probe Contamination

Sample probe contamination is checked between each sample by drawing ambient air through the probe via a Gilian pump and checking the response of the HNU PI 101. If HNU readings are higher than background, replacement or decontamination is necessary.

Sample probes may be decontaminated simply by drawing ambient air through the probe until the HNU reading is at background. More persistent contamination can be washed out using methanol and water, then air drying. Having more than one probe per sample team will reduce lag times between sample stations while probes are decontaminated.

3.9.4 Sample Train Contamination

The Teflon line forming the sample train from the probe to the Tedlar bag should be changed on a daily basis. If visible contamination (soil or water) is drawn into the sampling train, it should be changed immediately. When sampling in highly contaminated areas, the sampling train should be purged with ambient air, via a Gilian pump, for approximately 30 seconds between each sample. After purging, the sampling train can be checked using an HNU, or other field monitoring device, to establish the cleanliness of the Teflon line.

3.9.5 Field Blank

Each cooler containing samples should also contain one Tedlar bag of ultra-zero grade air, acting as a field blank. The field blank should accompany the samples in the field (while being collected) and when they are delivered for analysis. A fresh blank must be provided to be placed in the empty cooler pending additional sample collection. One new field blank per cooler of samples is required. A chain of custody form must accompany each cooler of samples and should include the blank that is dedicated to that group of samples.

3.9.6 Trip Standard

Each cooler containing samples should contain a Tedlar bag of standard gas to calibrate the

analytical instruments (Photovac GC, etc.). This trip standard will be used to determine any changes in concentrations of the target compounds during the course of the sampling day (e.g., migration through the sample bag, degradation, or adsorption). A fresh trip standard must be provided and placed in each cooler pending additional sample collection. A chain of custody form should accompany each cooler of samples and should include the trip standard that is dedicated to that group of samples.

3.9.7 Tedlar Bag Check

Prior to use, one bag should be removed from each lot (case of 100) of Tedlar bags to be used for sampling and checked for possible contamination as follows: the test bag should be filled with ultra-zero grade air; a sample should be drawn from the bag and analyzed via Photovac GC or whatever method is to be used for sample analysis. This procedure will ensure sample container cleanliness prior to the start of the sampling effort.

3.9.8 SUMMA Canister Check

From each lot of four cleaned SUMMA canisters, one is to be removed for a GC/MS certification check. If the canister passes certification, then it is re-evacuated and all four canisters from that lot are available for sampling.

If the chosen canister is contaminated, then the entire lot of four SUMMA canisters must be recleaned, and a single canister is re-analyzed by GC/MS for certification.

3.9.9 Options

Duplicate Samples

A minimum of 5% of all samples should be collected in duplicate (i.e., if a total of 100 samples are to be collected, five samples should be duplicated). In choosing which samples to duplicate, the following criterion applies: if, after filling the first Tedlar bag, and, evacuating the well for 15 seconds, the second HNU (or other field monitoring device being used) reading matches or is close to (within 50%) the first reading, a duplicate sample may be taken.

Spikes

A Tedlar bag spike and Tenax tube spike may be desirable in situations where high concentrations of contaminants other than the target compounds are found to exist (landfills, etc.). The additional level of QA/QC attained by this practice can be useful in determining the effects of interferences caused by these non-target compounds. SUMMA canisters containing samples are not spiked.

3.10 DATA VALIDATION

For each target compound, the level of concentration found in the sample must be greater than three times the level (for that compound) found in the field blank which accompanied that sample to be considered valid. The same criteria apply to target compounds detected in the Tedlar bag pre-sampling contamination check.

3.11 HEALTH AND SAFETY

Because the sample is being drawn from underground, and no contamination is introduced into the breathing zone, soil gas sampling usually occurs in Level D, unless the sampling location is within the hot zone of a site, which requires Level B or Level C protection. However, to ensure that the proper level of protection is utilized, constantly monitor the ambient air using the HNU PI 101 to obtain background readings during the sampling procedure. As long as the levels in ambient air do not rise above background, no upgrade of the level of protection is needed.

Also, perform an underground utility search prior to sampling (see section 3.4.4). When working with potentially hazardous materials, follow U.S. EPA, OSHA, and specific health and safety procedures.

4.0 General Surface Geophysics: SOP #2159

4.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the general procedures used to acquire surface geophysical data. This data is used for delineating subsurface waste, and for interpreting geologic, hydrogeologic or other data related to hazardous waste site characterization.

The media pertinent to these surface geophysical methods are soil/rock and groundwater. The sensitivity or minimum response of a given method depends on the comparison of the object or area of study to that of its background (i.e., what the media's response would be like without the object of study). Therefore, the suitability of surface geophysical methods for a given investigation must be judged on the object's ability to be measured and the extent to which the specific setting of the study interferes with the measurement.

The surface geophysical method(s) selected for application at a site are dependent on site conditions, such as depth to bedrock, depth to target, urban disturbances (fences, power lines, surface debris, etc.) and atmospheric conditions. Detectability of the target is dependent on the sensitivity of the instrument and the variation of the field measurement from the ambient noise. Ambient noise is the pervasive noise associated with an environment. Therefore, the applicability of geophysical methods at a given site is dependent on the specific setting at that site.

Five geophysical methods may be utilized in hazardous waste site characterization: magnetometry, electromagnetics, resistivity, seismology and ground penetrating radar (GPR). Magnetometers may be used to locate buried ferrous metallic objects and geologic information. Electromagnetic methods can be used to determine the presence of metals, electrical conductivity of the terrain, and geologic information. Resistivity methods are used to determine the electrical resistivity of the terrain and geologic information. Seismic methods are useful in determining geologic stratigraphy and structure. GPR may be used to locate disturbance in the soil (i.e., trenches, buried utilities and fill boundaries) and some near-surface geologic information.

These procedures may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the procedures employed should be documented and associated with the final report.

4.2 METHOD SUMMARY

4.2.1 Magnetics

A magnetometer is an instrument which measures magnetic field strength in units of gammas (nanoteslas). Local variations, or anomalies, in the earth's magnetic field are the result of disturbances caused mostly by variations in concentrations of ferromagnetic material in the vicinity of the magnetometer's sensor. A buried ferrous object, such as a steel drum or tank, locally distorts the earth's magnetic field and results in a magnetic anomaly. The objective of conducting a magnetic survey at a hazardous waste or groundwater pollution site is to map these anomalies and delineate the area containing buried sources of the anomalies.

Analysis of magnetic data can allow an experienced geophysicist to estimate the areal extent of buried ferrous targets, such as a steel tank or drum. Often, areas of burial can be prioritized upon examination of the data, with high priority areas indicating a near certainty of buried ferrous material. In some instances, estimates of depth of burial can be made from the data. Most of these depth estimates are graphical methods of interpretation, such as slope techniques and half-width rules, as described by Nettleton (1976). The accuracy of these methods is dependent upon the quality of the data and the skill of the interpreting geophysicist. An accuracy of 10 to 20 percent is considered acceptable. The magnetic method may also be used to map certain geologic features, such as igneous intrusions, which may play an important role in the hydrogeology of a groundwater pollution site.

Advantages

Advantages of using the magnetic method for the initial assessment of hazardous waste sites are the

relatively low cost of conducting the survey and the relative ease of completing a survey in a short amount of time. Little, if any, site preparation is necessary. Surveying requirements are not as stringent as for other methods and may be completed with a transit or Brunton-type pocket transit and a non-metallic measuring tape. Often, a magnetic investigation is a very cost-effective method for initial assessment of a hazardous waste site where buried steel drums or tanks are a concern.

Disadvantages

"Cultural noise" is a limitation of the magnetic method in certain areas. Man-made structures that are constructed with ferrous material, such as steel, have a detrimental effect on the quality of the data. Avoid features such as steel structures, power lines, metal fences, steel reinforced concrete, pipelines and underground utilities. When these features are unavoidable, note their locations in a field notebook and on the site map.

Another limitation of the magnetic method is the inability of the interpretation methods to differentiate between various steel objects. For instance, it is not possible to determine if an anomaly is the result of a steel tank, or a group of steel drums, or old washing machines. Also, the magnetic method does not allow the interpreter to determine the contents of a buried tank or drum.

4.2.2 Electromagnetics

The electromagnetic method is a geophysical technique based on the physical principles of inducing and detecting electrical current flow within geologic strata. A receiver detects these induced currents by measuring the resulting time-varying magnetic field. The electromagnetic method measures bulk conductivity (the inverse of resistivity) of geologic materials beneath the transmitter and receiver coils. Electromagnetics should not be confused with the electrical resistivity method. The difference between the two techniques is in the method which the electrical currents are forced to flow in the earth. In the electromagnetic method, currents are induced by the application of time-varying magnetic fields, whereas in the electrical resistivity method, current is injected into the ground through surface electrodes.

Electromagnetics can be used to locate pipes, utility

lines, cables, buried steel drums, trenches, buried waste, and concentrated contaminant plumes. The method can also be used to map shallow geologic features, such as lithologic changes and fault zones.

Advantages

Electromagnetic measurements can be collected rapidly and with a minimum number of field personnel. Most electromagnetic equipment used in groundwater pollution investigations is lightweight and easily portable. The electromagnetic method is one of the more commonly used geophysical techniques applied to groundwater pollution investigations.

Disadvantages

The main limitation of the electromagnetic method is "cultural noise". Sources of "cultural noise" can include: large metal objects, buried cables, pipes, buildings, and metal fences.

The electromagnetic method has limitations in areas where the geology varies laterally. These can cause conductivity anomalies or lineations, which might be misinterpreted as contaminant plumes.

4.2.3 Electrical Resistivity

The electrical resistivity method is used to map subsurface electrical resistivity structure, which is in turn interpreted by the geophysicist to determine the geologic structure and/or physical properties of the geologic materials. Electrical resistivities of geologic materials are measured in ohm-meters, and are functions of porosity, permeability, water saturation and the concentration of dissolved solids in the pore fluids.

Resistivity methods measure the bulk resistivity of the subsurface, as do the electromagnetic methods. The difference between the two methods is in the way that electrical currents are forced to flow in the earth. In the electrical resistivity method, current is injected into the ground through surface electrodes, whereas in electromagnetic methods currents are induced by application of time-varying magnetic fields.

Advantages

The principal advantage of the electrical resistivity method is that quantitative modeling is possible

using either computer software or published master curves. The resulting models can provide accurate estimates of depths, thicknesses and resistivities of subsurface layers. The layer resistivities can then be used to estimate the resistivity of the saturating fluid, which is related to the total concentration of dissolved solids in the fluid.

Disadvantages

The limitations of using the resistivity method in groundwater pollution site investigations are largely due to site characteristics, rather than in any inherent limitations of the method. Typically, polluted sites are located in industrial areas that contain an abundance of broad spectrum electrical noise. In conducting a resistivity survey, the voltages are relayed to the receiver over long wires that are grounded at each end. These wires act as antennae receiving the radiated electrical noise that in turn degrades the quality of the measured voltages.

Resistivity surveys require a fairly large area, far removed from pipelines and grounded metallic structures such as metal fences, pipelines and railroad tracks. This requirement precludes using resistivity on many polluted sites. However, the resistivity method can often be used successfully off-site to map the stratigraphy of the area surrounding the site. A general "rule of thumb" for resistivity surveying is that grounded structures be at least half of the maximum electrode spacing distance away from the axis of the survey line.

Another consideration in the resistivity method is that the fieldwork tends to be more labor intensive than some other geophysical techniques. A minimum of two to three crew members are required for the fieldwork.

4.2.4 Seismic

Surface seismic techniques used in groundwater pollution site investigations are largely restricted to seismic refraction and seismic reflection methods. The equipment used for both methods is fundamentally the same and both methods measure the travel-time of acoustic waves propagating through the subsurface. In the refraction method, the travel-time of waves refracted along an acoustic interface is measured, and in the reflection method, the travel-time of a wave which reflects or echoes off an interface is measured.

The interpretation of seismic data will yield subsurface velocity information, which is dependent upon the acoustic properties of the subsurface material. Various geologic materials can be categorized by their acoustic properties or velocities. Depth to geologic interfaces are calculated using the velocities obtained from a seismic investigation. The geologic information gained from a seismic investigation is then used in the hydrogeologic assessment of a groundwater pollution site and the surrounding area. The interpretation of seismic data indicates changes in lithology or stratigraphy, geologic structure, or water saturation (water table). Seismic methods are commonly used to determine the depth and structure of geologic and hydrogeologic units, to estimate hydraulic conductivity, to detect cavities or voids, to determine structure stability, to detect fractures and fault zones, and to estimate ripability. The choice of method depends upon the information needed and the nature of the study area. This decision must be made by a geophysicist who is experienced in both methods, is aware of the geologic information needed by the hydrogeologist, and is also aware of the environment of the study area. The refraction technique has been used more often than the reflection technique for hazardous waste site investigations.

Seismic Refraction Method

Seismic refraction is most commonly used at sites where bedrock is less than 500 feet below the ground surface. Seismic refraction is simply the travel path of a sound wave through an upper medium and along an interface and then back to the surface. A detailed discussion of the seismic refraction technique can be found in Dobrin (1976), Telford, et. al. (1985), and Musgrave (1967).

Advantages: Seismic refraction surveys are more common than reflection surveys for site investigations. The velocities of each layer can be determined from refraction data, and a relatively precise estimate of the depth to different interfaces can be calculated.

Refraction surveys add to depth information in-between boreholes. Subsurface information can be obtained between boreholes at a fraction of the cost of drilling. Refraction data can be used to determine the depth to the water table or bedrock. In buried valley areas, refraction surveys map the depth to bedrock. The velocity information

obtained from a refraction survey can be related to various physical properties of the bedrock. Rock types have certain ranges of velocities and these velocities are not always unique to a particular rock type. However, they can allow a geophysicist to differentiate between certain units, such as shales and granites.

Disadvantages: The seismic refraction method is based on several assumptions. To successfully resolve the subsurface using the refraction method, the conditions of the geologic environment must approximate these assumptions:

- the velocities of the layers increase with depth,
- the velocity contrast between layers is sufficient to resolve the interface, and
- the geometry of the geophones in relation to the refracting layers will permit the detection of thin layers.

These conditions must be met for accurate depth information.

Collecting and interpreting seismic refraction data has several disadvantages. Data collection can be labor intensive. Also, large line lengths are needed; therefore, as a general rule, the distance from the shot, or seismic source, to the first geophone station must be at least three times the desired depth of exploration.

Seismic Reflection Method

The seismic reflection method is not as commonly used on groundwater pollution site investigations as seismic refraction. In the seismic reflection method, a sound wave travels down to a geologic interface and reflects back to the surface. Reflections occur at an interface where there is a change in the acoustic properties of the subsurface material.

Advantages: The seismic reflection method yields information that allows the interpreter to discern between fairly discrete layers, so it is useful for mapping stratigraphy. Reflection data is usually presented in profile form, and depths to interfaces are represented as a function of time. Depth information can be obtained by converting time sections into depth measurements using velocities obtained from seismic refraction data, sonic logs, or

velocity logs. The reflection technique requires much less space than refraction surveys. The long offsets of the seismic source from the geophones, common in refraction surveys, are not required in the reflection method. In some geologic environments, reflection data can yield acceptable depth estimates.

Disadvantages: The major disadvantage to using reflection data is that a precise depth determination cannot be made. Velocities obtained from most reflection data are at least 10% and can be 20% of the true velocities. The interpretation of reflection data requires a qualitative approach. In addition to being more labor intensive, the acquisition of reflection data is more complex than refraction data.

The reflection method places higher requirements on the capabilities of the seismic equipment. Reflection data is commonly used in the petroleum exploration industry and requires a large amount of data processing time and lengthy data collection procedures. Although mainframe computers are often used in the reduction and analysis of large amounts of reflection data, recent advances have allowed for the use of personal computers on small reflection surveys for engineering purposes. In most cases, the data must be recorded digitally or converted to a digital format, to employ various numerical processing operations. The use of high resolution reflection seismic methods relies heavily on the geophysicist, the computer capacity, the data reduction and processing programs, resolution capabilities of the seismograph and geophones, and the ingenuity of the interpreter. Without these capabilities, reflection surveys are not recommended.

4.2.5 Ground Penetrating Radar

The ground penetrating radar (GPR) method is used for a variety of civil engineering, groundwater evaluation and hazardous waste site applications. This geophysical method is the most site-specific of all geophysical techniques, providing subsurface information ranging in depth from several tens of meters to only a fraction of a meter. A basic understanding of the function of the GPR instrument, together with a knowledge of the geology and mineralogy of the site, can help determine if GPR will be successful in the site assessment. When possible, the GPR technique should be integrated with other geophysical and

geologic data to provide the most comprehensive site assessment.

The GPR method uses a transmitter that emits pulses of high-frequency electromagnetic waves into the subsurface. The transmitter is either moved slowly across the ground surface or moved at fixed station intervals. The penetrating electromagnetic waves are scattered at points of change in the complex dielectric permittivity, which is a property of the subsurface material dependent primarily upon the bulk density, clay content and water content of the subsurface (Olhoeft, 1984). The electromagnetic energy which is scattered back to the receiving antenna on the surface is recorded as a function of time.

Depth penetration is severely limited by attenuation of the transmitted electromagnetic waves into the ground. Attenuation is caused by the sum of electrical conductivity, dielectric relaxation, and geometric scattering losses in the subsurface. Generally, penetration of radar frequencies is minimized by a shallow water table, an increase in the clay content of the subsurface, and in environments where the electrical resistivity of the subsurface is less than 30 ohm-meters (Olhoeft, 1986). Ground penetrating radar works best in dry sandy soil above the water table. At applicable sites, depth resolution should be between 1 and 10 meters (Benson, 1982).

The analog plot produced by a continuously recording GPR system is analogous to a seismic reflection profile; that is, data is represented as a function of horizontal distance versus time. This representation should not be confused with a geologic cross section which represents data as a function of horizontal distance versus depth. Because very high-frequency electromagnetic waves in the megahertz range are used by radar systems, and time delays are measured in nanoseconds (10^{-9} seconds), very high resolution of the subsurface is possible using GPR. This resolution can be as high as 0.1 meter. For depth determinations, it is necessary to correlate the recorded features with actual depth measurements from boreholes or from the results of other geophysical investigations. When properly interpreted, GPR data can optimally resolve changes in soil horizons, fractures, water insoluble contaminants, geological features, man-made buried objects, and hydrologic features such as water table depth and wetting fronts.

Advantages

Most GPR systems can provide a continuous display of data along a traverse which can often be interpreted qualitatively in the field. GPR is capable of providing high resolution data under favorable site conditions. The real-time capability of GPR results in a rapid turnaround, and allows the geophysicist to quickly evaluate subsurface site conditions.

Disadvantages

One of the major limitations of GPR is the site-specific nature of the technique. Another limitation is the cost of site preparation which is necessary prior to the survey. Most GPR units are towed across the ground surface. Ideally, the ground surface should be flat, dry, and clear of any brush or debris. The quality of the data can be degraded by a variety of factors, such as an uneven ground surface or various cultural noise sources. For these reasons, it is mandatory that the site be visited by the project geophysicist before a GPR investigation is proposed. The geophysicist should also evaluate all stratigraphic information available, such as borehole data and information on the depth to water table in the survey area.

4.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

This section is not applicable to this SOP.

4.4 INTERFERENCES AND POTENTIAL PROBLEMS

See section 4.2.1 for a discussion of limitations of the magnetic method.

See section 4.2.2 for a discussion of limitations of the electromagnetic method.

See section 4.2.3 for a discussion of limitations of the electrical resistivity method.

See section 4.2.4 for a discussion of limitations of the seismic refraction method and the seismic reflection method.

See section 4.2.5 for a discussion of limitations of the ground penetrating radar (GPR) method.

4.5 EQUIPMENT/APPARATUS

4.5.1 Magnetics

- GEM GSM-19G magnetometer/gradiometer, EDA OMNI IV magnetometer/gradiometer, Geonics 856AGX (with built-in datalogger) or equivalent
- magnetometer base station
- 300-foot tape measure
- non-ferrous survey stakes (wooden or plastic)

4.5.2 Electromagnetics

- Geonics EM-31, EM-34 or equivalent
- Polycorder datalogger
- Dat 31Q software (data dump software)
- 300-foot tape measure
- survey stakes

4.5.3 Electrical Resistivity

- DC resistivity unit (non-specific)
- 4 electrodes and appropriate cables (length dependent on depth of survey)
- 1 or 2 12-volt car batteries
- 300-foot tape measure

4.5.4 Seismic

- 12- or 24-channel seismograph (Geometrics 2401 or equivalent)
- 30 10Hz to 14Hz geophones (for refraction)
- 30 50Hz or greater geophones (for reflection)
- 300-foot tape measure
- survey stakes
- sledge hammer and metal plate or explosives

4.5.5 Ground Penetrating Radar

- GSSI SIR-8 or equivalent
- 80 Mhz, 100 Mhz or 300 Mhz antenna/receiver pit
- 200-foot cable
- 300-foot tape measure

4.6 REAGENTS

This section is not applicable to this SOP.

4.7 PROCEDURES

Refer to the manufacturer's operating manual for specific procedures relating to operation of the equipment.

4.8 CALCULATIONS

Calculations vary based on the geophysical method employed. Refer to the instrument-specific users manual for specific formulae.

4.9 QUALITY ASSURANCE/ QUALITY CONTROL

The following general quality assurance activities apply to the implementation of these procedures.

- All data must be documented on field data sheets or within site logbooks.
- All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

Method-specific quality assurance procedures may be found in the user's manual.

4.10 DATA VALIDATION

Evaluate data as per the criteria established in section 4.9 above.

4.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA and specific health and safety procedures.

APPENDIX A

Figures

Figure 1: Sampling Augers

SOP #2012

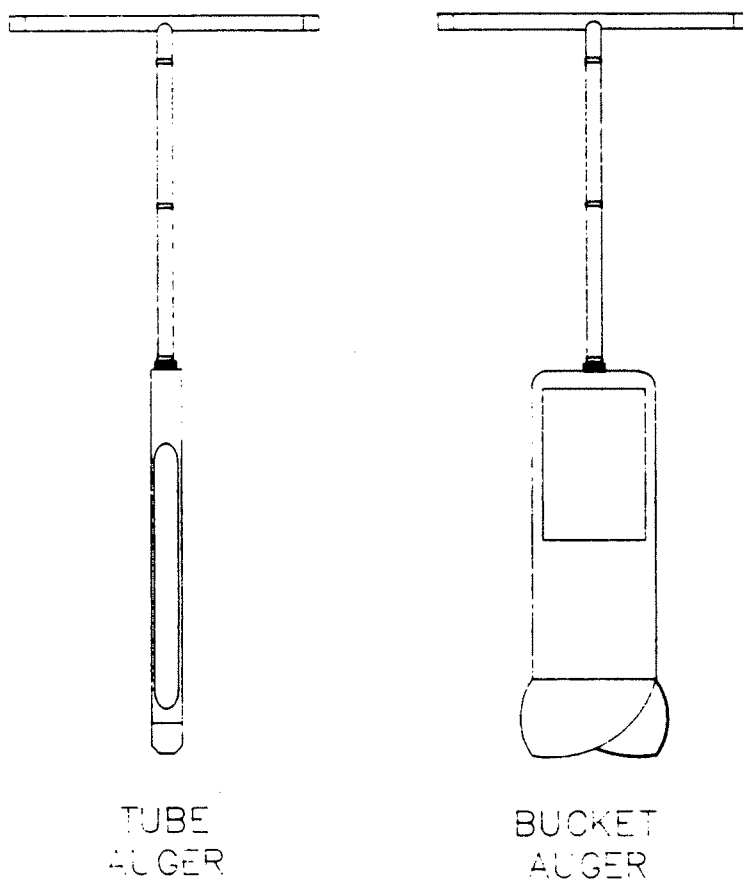


Figure 2: Sampling Trier

SOP #2012

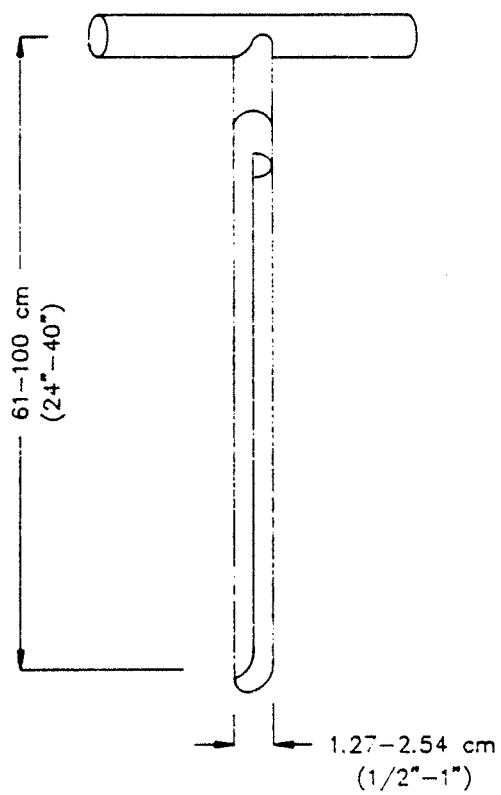
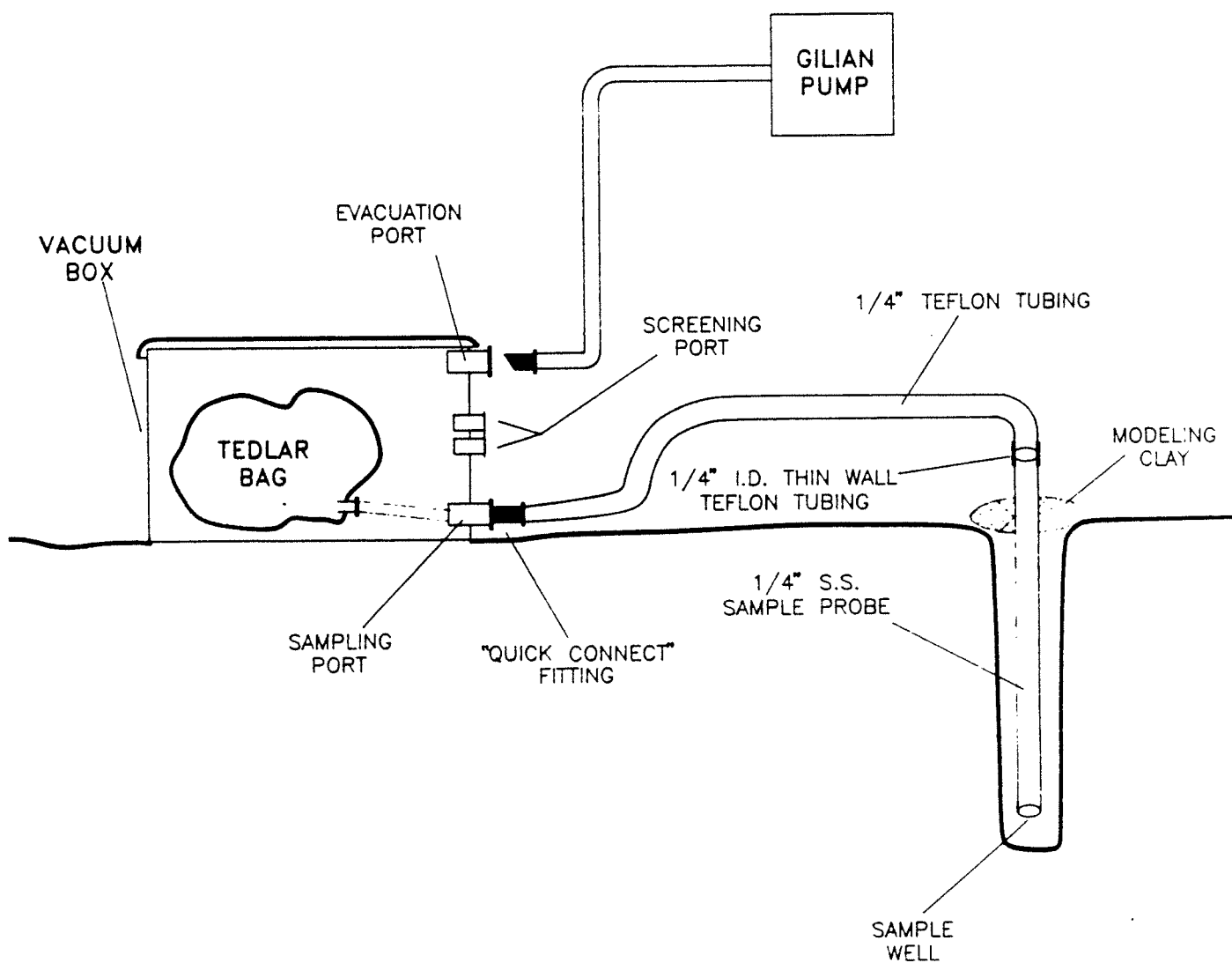


Figure 3: Sampling Train Schematic

SOP #2149



APPENDIX B

HNU Field Protocol

HNU Field Protocol SOP #2149

Startup Procedure

1. Before attaching the probe, check the function switch on the control panel to ensure that it is in the "off" position. Attach the probe by plugging it into the interface on the top of the readout module. Use care in aligning the prongs in the probe cord with the socket: do not force it.
2. Turn the function switch to the battery check position. The needle on the meter should read within or above the green area on the scale. If not, recharge the battery. If the red indicator light comes on, the battery needs recharging.
3. Turn the function switch to any range setting. For no more than 2 to 3 seconds, look into the end of the probe to see if the lamp is on. If it is on, you will see a purple glow. Do not stare into the probe any longer than three seconds. Long term exposure to UV light can damage the eyes. Also, listen for the hum of the fan motor.
4. To zero the instrument, turn the function switch to the standby position and rotate the zero adjustment until the meter reads zero. A calibration gas is not needed since this is an electronic zero adjustment. If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted, if necessary. Wait 15 to 20 seconds to ensure that the zero reading is stable. If necessary, readjust the zero.
2. Set the function switch to the range setting for the concentration of the calibration gas.
3. Attach a regulator (HNU 101-351) to a disposable cylinder of isobutylene gas. Connect the regulator to the probe of the HNU with a piece of clean Tygon tubing. Turn the valve on the regulator to the "on" position.
4. After 15 seconds, adjust the span dial until the meter reading equals the concentration of the calibration gas used. The calibration gas is usually 100 ppm of isobutylene in zero air. The cylinders are marked in benzene equivalents for the 10.2 eV probe (approximately 55 ppm benzene equivalent) and for the 11.7 eV probe (approximately 65 ppm benzene equivalent). Be careful to unlock the span dial before adjusting it. If the span has to be set below 3.0 calibration, the lamp and ion chamber should be inspected and cleaned as appropriate. For cleaning of the 11.7 eV probe, only use an electronic-grade, oil-free freon or similar water-free, grease-free solvent.
5. Record in the field log: the instrument ID # (EPA decal or serial number if the instrument is a rental); the initial and final span settings; the date and time; concentration and type of calibration used; and the name of the person who calibrated the instrument.

Operational Check

1. Follow the startup procedure.
2. With the instrument set on the 0-20 range, hold a solvent-based Magic Marker near the probe tip. If the meter deflects upscale, the instrument is working.

Field Calibration Procedure

1. Follow the startup procedure and the operational check.
4. When the activity is completed or at the end of the day, carefully clean the outside of the HNU with a damp disposable towel to remove any

Operation

1. Follow the startup procedure, operational check, and calibration check.
2. Set the function switch to the appropriate range. If the concentration of gases or vapors is unknown, set the function switch to the 0-20 ppm range. Adjust it as necessary.
3. While taking care not to permit the HNU to be exposed to excessive moisture, dirt, or contamination, monitor the work activity as specified in the site health and safety plan.

visible dirt. Return the HNU to a secure area and place on charge.

5. With the exception of the probe's inlet and exhaust, the HNU can be wrapped in clear

plastic to prevent it from becoming contaminated and to prevent water from getting inside in the event of precipitation.

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